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Textile Warp Size Reclamation Using Thermal Precipitation

Auburn Univ., AL

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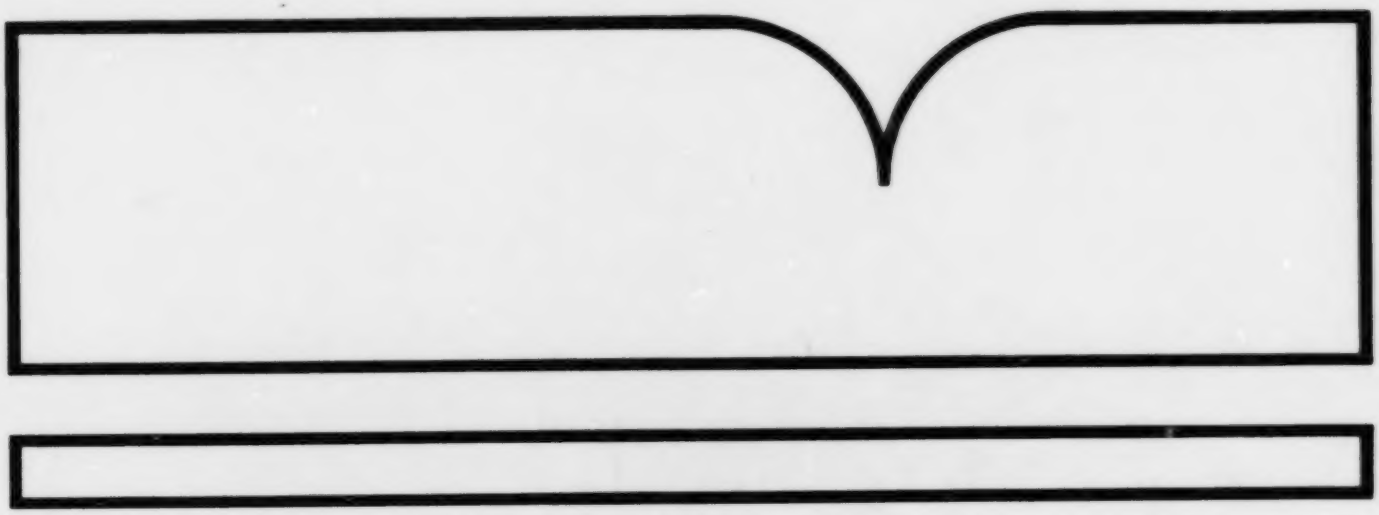

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16. ABSTRACT The report describes a novel method for reclaiming size based on precipitation of the size material by heating the desize washwater. The method uses hydroxypropyl cellulose (HPC) or hydroxypropyl methylcellulose (HPMC) which are soluble in cool water but insoluble in warm water. Reclaiming size would eliminate most of the BOD typically resulting from sizing and desizing. Since maximum system temperature with HPC is about 50 C, compared to 95 C for a conventional system, considerable energy is conserved. Strength and elongation of yarns sized with virgin HPC and with reclaimed HPC were not significantly different from those of yarns sized with the conventional size, polyvinyl alcohol (PVA), at similar add-on levels. Abrasion resistence of yarns sized with HPC was lower than that of yarns sized with PVA at similar add-on levels. Weaving of HPC-sized 50/50 polyester/cotton yarns in the laboratory was successful. In production weaving of percale sheeting, HPC accumulated on the shuttle caused poor weaving efficiency. The capital investment for reclaiming HPC using thermal precipitation is much lower than for reclaiming PVA using ultrafiltration. Operating costs for recovering HPC by thermal precipitation and for recovering PVA by ultrafiltration represent a savings over conventional sizing and desizing without reclaiming size.

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Cellulose Ethers	Stationary Sources	05C,11E
Textile Industry	Thermal Precipitation	13H
Textile Finishing	Washwater	
Sizing (Surface Treatment)	Hydroxypropyl Cellulose	14G
Reclamation	Hydroxypropyl Methyl-cellulose	07D
Precipitation (Chemstry)		
Water Treatment		

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TEXTILE WARP SIZE RECLAMATION USING THERMAL PRECIPITATION

BY

WARREN S. PERKINS, ROBERT P. WALKER AND LEO J. HIRTH
AUBURN UNIVERSITY, AUBURN, ALABAMA 36830

EPA GRANT NO. R 805128

PROJECT OFFICER
MAX SAMFIELD
INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

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ABSTRACT

A novel method for size reclamation based on precipitation of the size material by heating the desize washwater is reported. The method uses hydroxypropylcellulose (HPC) or hydroxypropylmethylcellulose (HPMC) which are soluble in cool water but insoluble in warm water. The HPC is applied to the yarn from a concentrated solution in cool water. The yarn is dried and woven into fabric. The fabric is desized using cool water to yield a dilute solution of the HPC. The desize washwater is heated to precipitate the HPC which is then separated from most of the water by settling, filtration or centrifugation to yield a concentrate suitable for reuse in slashing and water which is suitable for reuse in desizing.

Size reclamation would eliminate most of the BOD typically resulting from sizing and desizing. Since the maximum temperature in the system using HPC is about 50°C (122°F) compared to 95°C (203°F) for a conventional system using polyvinyl alcohol (PVA), starch or other conventional size materials, considerable energy is conserved.

Properties of films of HPC and HPMC are compared to properties of the conventional size, PVA. Strength and elongation of yarns sized with virgin HPC and with reclaimed HPC were not significantly different from that of yarns sized with PVA at similar add-on levels. Abrasion resistance of yarns sized with HPC was lower than that of yarns sized with PVA at similar add-on levels.

Weaving of HPC-sized 50/50 polyester/cotton yarns in the laboratory was successful. In production weaving of percale sheeting the HPC accumulated on the shuttle and caused poor weaving efficiency.

The capital investment for reclamation of HPC using thermal precipitation is much lower than that for reclamation of PVA using ultrafiltration. The operating costs for recovery of HPC by thermal precipitation and for recovery of PVA by ultrafiltration represents a savings over conventional sizing and desizing without size reclamation. The operating costs for reclamation of HPC are slightly higher than for reclamation of PVA, but the pay back period for an HPC reclamation system is estimated to be 2.04 years compared to 3.74 years for an ultrafiltration system for PVA reclamation.

This report was submitted in fulfillment of EPA Grant Number R805128 by the Auburn University Department of Textile Engineering under the partial sponsorship of the U. S. Environmental Protection Agency. The work was performed in cooperation with the Alabama Textile Education Foundation through the Auburn University Water Resources Research Institute. This report covers the period from March 21, 1977 to December 20, 1979 and work was completed as of December 20, 1979.

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ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

BOD	--	Biochemical Oxygen Demand
CMC	--	carboxymethylcellulose
CMS	--	carboxymethylstarch
cm	--	centimeter (s)
COD	--	Chemical Oxygen Demand
HEC	--	hydroxyethylcellulose
HPC	--	hydroxypropylcellulose
HPMC	--	hydroxypropylmethylcellulose
in	--	inch(es)
mil	--	0.001 inch
min	--	minutes
oz	--	ounce(s)
PE	--	polyester
psi	--	pounds per square inch
PVA	--	polyvinyl alcohol
sec	--	second(s)

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Several chemical companies supplied polymers and other chemicals at no cost to the project. Among these chemical suppliers were Hercules, Inc. and Dow Chemical Company. This support is gratefully acknowledged.

The mill work was done in cooperation with West Point Pepperell, Inc. Their willingness to make their facilities available at their research center and in their plants made much of this work possible. This excellent support is appreciated and acknowledged.

Thanks are also expressed to Dr. Max Samfield, Project Officer and Mr. Jack Kernan of EPA for their interest in the work and for their assistance in all stages of the work.

SECTION 1

INTRODUCTION

Virtually all textile yarns which are to be woven into the lengthwise direction (warp) in a fabric are coated with a protective material, called size, during the slashing process. The purpose of this size is to improve the performance of the yarn in the weaving process. In most cases the size material is removed from the woven fabric in the finishing plant. Published data indicate that 45% to 75% of the BOD load from a woven fabrics finishing plant results from these warp sizes which are removed from the fabric.

A process to recover and recycle the warp size used in textile processes has the potential to lower the BOD and COD of textile finishing plant effluents more than any other single change in textile processing. Such a process has been the objective of much development work in recent years. The following are specific examples of approaches to size recovery and recycle:

a) Recovery of carboxymethyl cellulose by chemical precipitation from the desize wastewater has been studied but not commercialized.

b) Recovery of polyvinyl alcohol (PVA) by chemical precipitation has also been subject to investigation.

c) Studies of recovery of warp sizes via solvent sizing and desizing have been made. A solvent sizing and solvent desizing system has been developed by two (2) Italian machinery companies.

d) Recovery of polyvinyl alcohol (PVA) by ultrafiltration of the desize wastewater has been subject to considerable development and commercial processes have now been operating for several years.

The tremendous interest of the U.S. Textile Industry in recycle of warp sizes is understandable since the economic outlook for such a development is as favorable as are the environmental considerations. The size material adds no value to the product (fabric) since it is normally removed in desizing and disposed of in a wastewater treatment facility. Therefore, the size material itself adds to the cost of fabric production. A process for recovery and reuse of size material may prove profitable to the textile mill through reduced fabric production cost if a high degree of recovery of the size material is obtained.

Therefore, a size reclamation process offers the potential for a modification from which the mill may realize a direct return on investment.

This approach should be considerably more attractive to management than the prospect of construction of additional treatment facilities which may become obsolete as the 1983 Effluent Guidelines are implemented. Further, size recycling will allow the industry room for expansion and growth without great capital investment for wastewater treatment facilities since recovery and recycle of warp sizes will allow increases in production without large increases in effluent loads.

The size reclamation system which is the subject of this report uses a size material selected from a group of polymers having the unusual property of being soluble in water at room temperature but insoluble at some higher temperature. Hydroxypropylcellulose (HPC) is such a polymer. HPC is soluble in water at temperatures below 43°C (109°F) but becomes almost completely insoluble in water at temperatures above 45°C (113°F). Hydroxypropylmethylcellulose (HPMC) also exhibits this "reverse solubility" property but at a higher precipitation temperature than is required for HPC. Most of the work done in this project was concerned with HPC rather than HPMC and except where otherwise specified the discussions pertain to HPC rather than HPMC.

Precipitation of HPC or HPMC from desize wastewater can be accomplished by heating the desize wastewater. A schematic diagram of the desizing and recovery system is shown in Figure 1. Application of the size material to the warp yarn in slashing and washing of the size material from the woven fabric in desizing is done with water at a temperature lower than 43°C. The desize washwater containing HPC at a concentration of 0.5 to 1.5% by weight is filtered to remove insoluble impurities and heated to above 45°C to precipitate the HPC. The precipitated HPC is then separated from most of the water. The HPC redissolves when cooled to yield a solution concentrated enough to reuse in slashing. The recovered water can be cooled to below 43°C and reused in the desize washer.

This report describes both laboratory and in-plant work concerned with the development of a process utilizing HPC as the warp size with the objective of reclaiming the HPC using the thermal precipitation principle. Work was performed in the following areas:

1) Utility and performance of HPC as a textile warp size.

2) Design and operation of a thermal precipitation size reclamation system.

3) Economics of size reclamation by thermal precipitation.

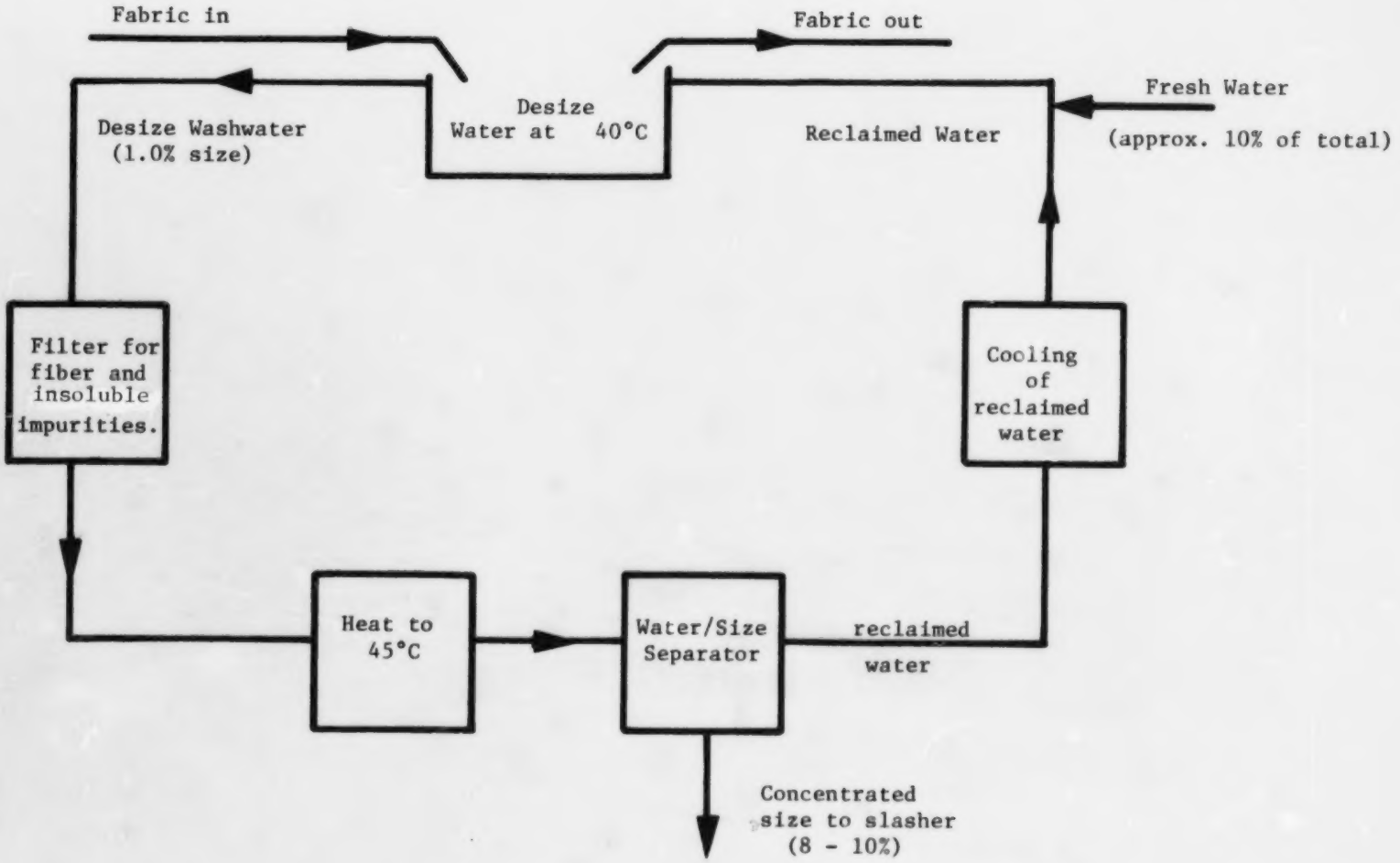

FIGURE 1. Schematic of desize and recovery system.

SECTION 2

CONCLUSIONS

Hydroxypropylcellulose (HPC) is soluble in water at room temperature but becomes virtually insoluble in water at a temperature greater than about 43°C. HPC can be precipitated from water by heating the water to above the precipitation temperature. The HPC can then be separated from most of the water by settling, filtration or centrifugation. Therefore, a dilute solution such as is obtainable in a conventional desize washer can be concentrated to the extent required for use of the material in a conventional slashing process. More than 90% of the HPC in a 1.0% solution of desize washwater can be obtained at up to 25% concentration in the reclaimed solution using a recovery system based on settling of the HPC in warm water. Hydroxypropylmethylcellulose (HPMC) also exhibits this thermal precipitation phenomenon but at a higher temperature, about 65°C.

Textile yarns consisting of 100% cotton or 50% polyester and 50% cotton can be sized with HPC or HPMC. The physical properties of yarns sized with HPC or HPMC are adequate for the yarns to be woven into fabric. The tensile strength of yarn sized with HPC is appreciably higher than that of unsized yarn and is not significantly different from the tensile strength of yarns sized with the conventional size polyvinyl alcohol (PVA) at the same size add-on. The abrasion resistance of yarns sized with HPC is higher than that of unsized yarns. The magnitude of the improvement in abrasion resistance depends on the size add-on level. The abrasion resistance of yarns sized with PVA is higher than that of yarns sized with HPC at the same add-on level.

Clear flexible films are formed upon evaporation of the water from aqueous solutions of HPC. Clear films are produced even when the temperature is higher than that which causes the HPC to precipitate from water. The strength of HPC films is similar to that of conventional sizes such as starch and carboxymethylcellulose. The elongation of films of HPC is intermediate between that of conventional sizes, starch and PVA.

Yarns sized with HPC can be woven into fabric. On 50-inch wide looms weaving at 215 filling insertions per minute, the contact of the shuttle with the yarn results in an accumulation of HPC on the shuttle. This accumulation of HPC on the shuttle is undesirable because it eventually interferes with the passage of the shuttle into the yarn shed causing excessive yarn breaks near the sides of the loom. The accumulated size material may also be periodically stripped away from the shuttle and become woven into the fabric causing a fabric defect. This accumulation of HPC on the shuttle does not occur to a disruptive degree on 24-inch looms weaving at 175 filling insertions per minute. Whether this accumulation of size on the

shuttle also occurs with HPMC on 50-inch looms is not known, but the accumulation of HPMC does not occur to a disruptive degree on 24-inch looms weaving at 175 filling insertions per minute.

HPC reclaimed from woven fabric has tensile strength and elongation not appreciably different from that of virgin HPC. Yarns sized with reclaimed HPC have abrasion resistance not appreciably different from that of similar yarns sized with virgin HPC. Pure HPC may be more flexible than is desirable in a size material. HPC softens dramatically at high relative humidity, and its effectiveness as a warp size deteriorates.

Fabrics containing HPC can be rapidly desized using room-temperature water. The rate of removal of HPC from a polyester/cotton fabric is as rapid at 25°C (77°F) as the removal of PVA at 95°C (203°F). Water used for desizing and subsequently separated from the HPC in recovery system is suitable for reuse in desizing. The content of impurities in desize washwater increases with each reuse cycle. A steady state level of impurities is reached if a fraction of the desize washwater is discharged at each cycle.

Thermally precipitated HPC particles have a negative zeta potential in water. Consequently, coagulation and settling of HPC particles is enhanced by addition of cations such as Ca^{++} or Al^{+++}. The addition of cations does not have an appreciable effect on the settling rate of particles of thermally precipitated HPMC.

Virgin HPC is resistant to biodegradation and solutions can be stored for long periods. Reclaimed HPC solutions are subject to biodegradation because of the presence of natural impurities removed from the fabric. Reclamined HPC solutions can be preserved with agents typically used for preservation of textile chemicals. HPC is stable for long periods at the temperature necessary for its reclamation.

The capital investment for reclamation of HPC by thermal precipitation is about one-third of that for reclamation of polyvinyl alcohol by ultrafiltration. The operating costs including size material costs for a system based on reclamation of HPC by thermal precipitation is slightly higher than for a system based on reclamation of PVA by ultrafiltration. The pay back period for a thermal precipitation system is about one-third of that for an ultrafiltration system. Because of the value of the reclaimed material, size reclamation systems based on thermal precipitation and ultrafiltration both operate at lower costs than a conventional desizing system not involving size reclamation.

A desize and reclamation system using HPC operates at a maximum water temperature of about 50° while a system using PVA operates at a temperature of about 95°C. The result is an energy savings of about 0.655×10^6 BTU's per 1000 pounds of fabric desized.

SECTION 3

RECOMMENDATIONS

Size reclamation is an economical approach to chemical and water conservation and will reduce the pollution load from most woven fabrics finishing plants. The size reclamation technique that is most applicable to a particular mill must be selected based on factors peculiar to that mill. A critical comparison of all size reclamation techniques is not presently available, but such a comparison would be useful for decision making people in the textile industry.

Size reclamation using thermal precipitation appears to be technically and economically feasible at present only for selected types of plants. Those plants weaving cotton or polyester/cotton on narrow looms can probably use hydroxypropyl cellulose as the warp size. This size can be recovered by thermal precipitation and reused for sizing. HPC may also be useful in shuttleless weaving. The possibility of using HPC as a size in machines using projectile, rapier and air jet filling insertions mechanisms should be investigated. HPC is not recommended for weaving of 50-inch wide fabrics at a speed of 215 picks per minute using shuttle looms at the current stage in its development as a warp size.

The greatest deficiency of HPC as a warp size is insufficient hardness. The possibility of chemical modification of the material to make this property suitable for warp sizing purposes should be investigated. This work would best be done by a chemical company experienced in evaluation of polymers for warp sizing.

HPC accumulates on the shuttle during the weaving of 50-inch wide fabrics if the shuttle is made of wood or of laminated cotton fabric composite materials. Shuttles made of other materials should be tested to determine if the accumulation of HPC occurs.

Hot melt sizing is a developing technology for the textile industry. The process applies a molten size to the yarn and uses no water. The first production model hot melt sizing machine will be available in the near future. Since HPC is a thermoplastic material its applicability as a hot melt size should be investigated. Conventional recoverable size materials such as PVA and CMC are not applicable by the hot melt technique and size materials which have been developed especially for hot melt sizing are not recoverable. Therefore, the availability of a recoverable hot melt size would be highly desirable.

SECTION 4

PROPERTIES OF HPC

INTRODUCTION

Hydroxypropylcellulose is a nonionic cellulose ether made by the reaction of cellulose with propylene oxide. HPC is soluble in water at temperatures below 38°C (100°F) and insoluble in water at temperatures above 45°C (113°F). HPC is also soluble in a number of relative polar organic solvents such as acetone and ethanol and it even has relatively high solubility in methylene chloride. HPC is phsiologically inert and the food grades have been cleared by the FDA as intentional food additives. HPC is thermoplastic having a softening temperature of 130°C. It is available in a wide variety of viscosity ranges. HPC has a number of specific applications some of which utilize its property of film-formation.

Hydroxypropylcellulose (HPC) is hygroscopic. It absorbs water from the atmosphere and becomes soft and extremely flexible at high relative humidity. Its equilibrium moisture content is 4% at 50% RH and 73°F and 12% at 84% RH and 73°F.

Virgin HPC in aqueous solution is resistant to microbiological degradation. This resistance to microbial attack decreases dramatically when the HPC solution contains impurities extracted from cotton. Giv-Guard DXN® is an effective preservative for solutions of HPC and was used for this purpose throughout this project. A number of other preservatives are also effective for HPC solutions.

HPC is surface active. Aqueous solutions have low surface tension which promotes foaming and air entrainment. Water dispersible defoamers are effective on dilute solutions, but the high viscosity of more concentrated solutions inhibits the release of entrained air.

Hydroxypropylmethylcellulose (HPMC) is a cellulose ether prepared by the reaction of alkali cellulose with methyl chloride and propylene oxide. HPMC is similar to HPC in some respects. It too is soluble in cool water but insoluble in hot water. HPMC precipitates at about 70°C (158°F) whereas HPC precipitates at about 43°C (109°F). HPMC is thermoplastic but its softening temperature, 240°C, is considerably higher than that of HPC. HPMC is less flexible and harder than HPC. HPMC is also soluble in certain relatively polar organic solvents.

EXPERIMENTAL PROCEDURES

Film Casting

Films were cast from 5.0% solutions of the polymer (size) in water. The solution was poured onto mercury in a rectangular dish or spread uniformly on glass plates to allow the water to evaporate and leave a solid film.

Tensile Strength

Strips of film 1.27 cm (1.0 inch) wide and of measured thickness were broken on an Instron Tensile Tester. The gauge length was 7.62 cm (3 inches) and the crosshead speed was 5.08 cm/minute (2 inches/minute).

Resolubility

The length of time necessary for a 0.00762 cm (3 mil) thick x 1.27 cm wide x 2.54 cm long strip of the film to dissolve in water at the test temperature was determined.

Adhesion

Strips of the fiber substrate (cellophane or mylar polyester) 2.54 cm (1 inch) wide by 17.78 cm (7 inches) long were used for the adhesion tests. One (1) drop of a 5.0% solution of the size material in water was placed on one end of the strip of film. A second strip of film was placed over the first strip with a 6.45 square cm (1 square inch) overlap. The strips were placed under 4.54 kilograms (10 pounds) of pressure for at least 12 hours at 50°C to dry. The force necessary to separate the strips was determined using an Instron Tensile Tester.

RESULTS AND DISCUSSION

Tensile and Adhesion

All size materials are film forming agents. It is generally accepted that physical properties of films of size materials can be used to predict the effect of the size on yarn physical properties. The physical properties of the yarn in turn affect the performance of the yarn in weaving. Size material film properties which affect the properties of the yarn to which they are applied include tensile strength, elongation, flexibility and adhesion to the fiber being sized. Each of these properties was measured using films of hydroxypropyl cellulose and compared to properties of films of polyvinyl alcohol (PVA) and carboxymethyl cellulose (CMC), typical aqueous warp sizes.

The size material must possess sufficient tensile strength to bond together the individual fibers in a yarn. However, the strength of the size material must be low enough to allow the yarns to be separated at the slasher split rods without causing yarn breakage. Polyvinyl alcohol is known to cause splitting problems in certain cases, and the add-on of polyvinyl alcohol to the yarn must be low if fine yarns are to be sized successfully. The

tensile strength and elongation of an individual film depends greatly on the conditions used to prepare the film as well as the film thickness. Therefore, the values reported by different laboratories often do not correlate well. The values reported in Table 1 were all measured in our laboratories and are comparable to each other although possibly not comparable to values reported in the literature or by suppliers of the materials.

The tensile strength of HPC is somewhat lower than that of the commonly used warp sizes PVA and CMC. On the other hand, the tensile strength of HPMC is somewhat higher than that of the PVA and CMC films cast in this study. The absolute values of tensile strength of all of these materials should be adequate for warp sizing. Use of an additive such as a lubricant or softener which would reduce the film strength of PVA, HPC and CMC would probably be desirable. However, such an additive would be undesirable with HPC since its strength is low enough that it should split easily at the slasher lease rods without the addition of softeners, etc.

Sufficient elongation of the size material is necessary if the material is to withstand the repeated cyclic stresses encountered by the yarn in weaving. If the size material ruptures when the yarn is stretched in weaving, the size material will become less tightly bound to the yarn and excessive shedding will result. A minimum elongation of 4 to 5% is required in a size material, but higher elongation is not essential. The elongation of HPC is of the same order of magnitude as that of polyvinyl alcohol while the elongation of HPMC is very similar to that of CMC. The elongation of either HPC or HPMC are adequate for warp sizing purposes.

A warp size must be easily removable from the woven fabric in desizing. The ease of removal of the size material is related to its solublity in the desizing medium. Polyvinyl alcohol is normally desized using large volumes of hot water. Hydroxypropylcellulose films dissolved as readily in water at 25°C as did polyvinyl alcohol in water at 95°C. Heating of films of hydroxypropylcellulose at 110°C for 6 hours had little effect on the ability of the film to dissolve in water.

Adhesion of the size material is imperative since the size must bond together the fibers in the yarn. Further, the size material must adhere to both fibers if the yarn being sized is a blend. Hydroxypropylcellulose adheres well to both polyester and cellulose. A certain degree of flexibility is desirable in the size film on the yarn since it must withstand repeated bending as the yarn passes through the weaving elements. HPC is considerably more flexible than PVA and CMC. However, pure HPC may be more flexible than is desirable in a size material. HPC softens dramatically at high relative humidity, and its effectiveness as a warp size deteriorates. Additives which harden or reduce the hygroscopic degree of HPC may prove beneficial in the size formulation. The use of additives to reduce the tendency of the size material to attract moisture is not practiced in warp sizing since conventional sizes normally need placticization to soften or increase the flexibility of the film rather than hardening..

TABLE 1. PROPERTIES OF HYDROXYPROPYLCELLULOSE, [a] HYDROXYPROPYLMETHYL-CELLULOSE[b] POLYVINYL ALCOHOL[c], AND CMC[d]

PROPERTY	MATERIAL			
	HPC	HPMC	PVA	CMC
Tensile Strength (psi)	800-1300	5000	3000	3500
Elongation at Break (%)	50	6-9	150-200	7
Resolubility of film[e]	Good	Good	Good	Excellent
Adhesion to cellophane (pounds)	high[f]	-	high[f]	-
Adhesion to mylar polyester (pounds)	15	-	13	-

[a] Hercules, Inc. Klucel J

[b] Dow Chemical Company, Experimental Polymer J-15

[c] DuPont, Elvanol T-25

[d] Hercules, Inc. Warp Size Grade CMC

[e] In water at 95°C for PVA and CMC and 25°C for HPC

[f] Adhesive bond was greater than the strength of the cellophane film which was about 12 pounds for the 1-inch wide strip used in the test.

Stability

The HPC must be heated to above 45°C (113°F) each time it is cycled through the recovery system. Therefore, the HPC must be relatively stable in water at the required temperature. A test was performed to ascertain if HPC degrades in aqueous medium upon prolonged heating. The viscosity of 1.0% solution of HPC in water at pH= 7.0 was measured at 30°C using a Ubbelohde viscometer. The solution was then heated to 58°C and kept at that temperature for 24 hours. The solution was then cooled to 30°C and the viscosity was measured again. There was no appreciable change in the viscosity of the HPC solution during this experiment indicating that the molecular weight (or degree of polymerization) did not change appreciably. Further, there were no other visually detectable changes in the solution during the test. It is important to note that HPC will undergo hydrolysis with decrease in degree of polymerization in either strongly acidic or strongly alkaline medium. According to the manufacturer, HPC solutions should be kept in the pH range of 6.0 to 8.0 to prevent degradation.

Drying Characteristics

Since hydroxypropylcellulose is not soluble in water at temperatures greater than 45-50°C, tests were performed to ascertain whether satisfactory films are formed from solutions of HPC in water when heat is used to vaporize the water. Film formation by the size material on the yarn is necessary to achieve good size performance.

Thin coatings of a 5.0% aqueous solution of hydroxypropylcellulose (HPC) were applied to glass plates. Individual plates were dried at different temperatures ranging from room temperature to 200°C. The appearance of the HPC during drying was observed and tensile properties of the resulting films were measured.

When the drying temperature was below 45°C, the HPC formed a clear film from the clear solution of HPC on the glass. At drying temperatures above 50°C, the HPC solution on the glass became cloudy, and a film formed over the top of the liquid. The solution dried gradually from edge to center of the glass plate leaving a clear film appearing virtually the same to the eye as the film deposited at temperatures below 45°C. However, the physical properties of the films deposited at high and low temperature were not the same. The films formed at 85°C had tensile strength of about 1500 psi and elongation of about 22%, while the films formed at room temperature had tensile strength of about 800 psi and elongation of about 42%. Therefore, high drying temperatures apparently do not prevent the HPC from forming films having good properties.

Effect of Additives

As discussed elsewhere in this report, an additive must be used to facilitate the settling of thermally precipitated HPC in water. Inorganic salts are effective in assisting the settling of HPC. If an additive is used to assist settling, the additive must not have a serious detrimental effect

on the properties of the size film on the yarn, or the performance of the yarn in weaving will deteriorate. HPC films were cast from solutions containing known quantities of various salts which can be used to assist settling. The data in Table 2 shows the properties of some of these films. Sodium chloride in the film at a level as low as 1% on the weight of the film caused the film to be weak and brittle. Calcium chloride in the film at a level of 1% on the weight of the film caused a decrease in the strength of the film. Films containing aluminum sulfate at a level of either 1% or 5% on weight of the HPC were as strong, flexible and stretchable as the HPC films which contained no additives. Aluminum sulfate appears to be a very desirable additive since it efficiently enhances the settling of thermally precipitated HPC and causes no apparent deterioration of the properties of HPC films.

Lubricants are commonly used in size formulations for textile yarns. However, the lubricants most commonly used in size formulations are not acceptable in a formulation based on HPC because a minimum temperature of 60°C (140°F) is required to disperse the lubricant. Further, water soluble lubricants are not generally considered desirable because they become mixed with the size film rather than migrating to the surface of the yarn where the lubricating effect is needed. A water dispersible liquid lubricant, Abco Chemical's Ester D-32, was tested in this project. As the data in Table 2 shows the product does cause a decrease in film strength and elongation of HPC. However, the effect is not dramatic and the product may impart desirable properties to yarns sized with HPC. Results of other tests relating to possible beneficial effects of Ester D-32 on yarn properties were inconclusive.

Hydroxypropylcellulose is extremely surface active and solutions of HPC in water have a marked tendency to foam. Since foam in the system can cause processing problems some work was done on defoamers that might be used in the HPC solutions. The Ester D-32 described as a lubricant in the previous paragraph markedly decreased the foaming tendency of HPC. Two (2) other defoamers that were effective are octyl alcohol and Union Carbide's SAG-10, a silicone type defoamer. As indicated in Table 2 SAG-10 at a level of 0.05% on weight of the HPC caused no deterioration in film properties.

Effect of Reclamation

Data on the tensile strength and elongation of reclaimed HPC is shown in Table 3. The HPC used to make the films for these tests was reclaimed by thermal precipitation of the HPC from the desize washwater obtained by washing fabric in a continuous open-width washer. The fabric was woven during the in-plant trials performed in the project. The strength of the reclaimed HPC was as high and possibly slightly higher than that of virgin HPC even though the reclaimed HPC obviously contains impurities removed from the fabric as evidenced by the pronounced brown coloration of the film as compared to the clear colorless film obtained with virgin HPC. The elongation of the reclaimed HPC is somewhat lower than that of the virgin HPC indicating that the impurities present in the reclaimed HPC did not plasticize the film appreciably. The retention of strength in the reclaimed HPC can be rationalized by the fact that some non-recoverable impurities are

TABLE 2 . EFFECT OF ADDITIVES ON PROPERTIES OF HPC FILMS.

Additive	Amount (% wt. of HPC)	Tensile Strength (psi)	Elongation at Break (%)
None (control)	---	900	70
Sodium Chloride	10	Film too weak to test	
Sodium choloride	1	Film was brittle, not tested	
Calcium chloride	1	400	90
Aluminum sulfate	5	950	73
Aluminum sulfate	1	900	72
Ester D-32	5	500	50
Ester D-32	3	650	57
Ester D-32	1	600	56
SAG-10	0.05	1000	56

present in the HPC as received from the manufacturer. As explained elsewhere in this report, these impurities are lost during the reclamation process. Therefore, the quality of the HPC may be improved by going through the reclamation process. Upon repeated use the concentration of impurities in HPC would probably increase to the point that the properties of the HPC would be adversely affected. The concentration of impurities would be controlled by either periodic or continuous discharge of a portion of the reclaimed water in lieu of returning all of the water to the desize washer.

Carbowax 20M was tested as an additive for size formulations based on HPC. Carbowax 20M is recommended as a hardening agent for HPC for other applications of HPC.[1] The effect of carbowax 20M on properties of films cast using reclaimed HPC is shown in Table 3. The presence of as little as 1% carbowax based on weight of HPC caused a dramatic loss in strength of the film. At the 5% level of carbowax 20M the loss in film strength was more than 50%. The presence of carbowax 20M did not have a large effect on breaking elongation of the HPC films.

TABLE 3. PROPERTIES OF FILMS OF RECLAIMED HPC.

Description	Film Strength (Psi)	Film Elongation (%)
Reclaimed HPC	1020	37
Reclaimed HPC, 1% Carbowax 20M	699	58
Reclaimed HPC, 5% Carbowax	488	30

Assessment of Hydroxypropylmethylcellulose (HPMC)

As stated previously HPMC was studied in this project as well as HPC. The majority of the work was done with HPC rather than HPMC for two reasons. First, HPC (Hercules, Inc. Klucel®) was the material available at the beginning of the project and was also the material upon which the project proposal was written. An HPMC product in the viscosity range required for warp sizing was not available at that time. Therefore, all of the work early in the project on properties of the materials was done with HPC. Later in the project when an HPMC in the proper viscosity range became available, the project schedule did not allow for its complete evaluation. Therefore, all of the in-plant work was done with HPC rather than HPMC.

Secondly, the HPMC products obtained from Dow Chemical Company for this project were all experimental polymers. They were prepared by acid hydrolysis of commercially available HPMC products to decrease the molecular weight to a range that would allow the preparation of a solution of the proper viscosity for warp sizing. The result was that the distribution of molecular weights in the product was such that a large portion of the product was not recoverable by thermal precipitation. According to Dow Chemical Company an HPMC product of the proper viscosity would be made commercially by degrading the cellulose to the proper molecular weight in the alkali ageing stage before the etherification reaction. Thereby the molecular weight distribution would be controlled. Less very short polymer chains would be produced, and the polymer would be more recoverable by thermal precipitation.

The rationale for this argument is good as indicated in thermal precipitation experiments performed in this project. A 10.0% solution of HPMC (product identified as J-15 grade) had viscosity of 1710 centipoise at 30°C. The viscosity of a 10% solution of HPMC (J-15) which had been reclaimed from water by thermal precipitation was 5440 centipoise at 30°C. The recovery level of this HPMC was about 60%. These figures indicate that the 40% of the HPMC which was not reclaimed by thermal precipitation consists of low molecular weight material. Further, when the reclaimed HPMC was subjected to a second cycle through the thermal precipitation recovery system the recovery level was higher than 98%. This means that an HPMC product not containing the low molecular weight fraction would be recoverable at a very high level by thermal precipitation.

Films of the HPMC products used in this work were stronger than films of HPC when the viscosity of the two materials was the same. HPMC is also harder, less flexible and less sensitive to moisture than is HPC. Therefore, HPMC may not be as susceptible to deterioration in performance at high humidity as is HPC. HPMC has a softening temperature of over 200°C as compared to the softening temperature of about 130°C for HPC. There is no proof that the tendency of HPC to the stick to and build up on the shuttle during weaving is related to softening of the HPC. However, it is possible that this problem is caused by softening of the HPC as the shuttle is heated by frictional contact with other loom parts. Weaving tests on a narrow loom indicated that HPMC does not have as much tendency to stick to the shuttle as does HPC. However, a plant trial was not run with the HPMC because a gradual accumulation of HPMC occurred on the slasher drying cylinders during application of the HPMC and there was an insufficient supply of the HPMC polymer. This accumulation was not a problem for short lab trials but would have to be overcome before production trials could be performed.

SECTION 5

PROPERTIES OF SIZED YARNS

INTRODUCTION

The performance of yarns in weaving depends on yarn physical properties such as tenacity, elongation at the break and abrasion resistance. The required level of these properties for good weaving performance depends on the type of yarn being processed.

The physical properties of 36's cotton counts, 50/50 polyester cotton yarns sized with the recoverable sizes, hydroxypropylcellulose (HPC) and hydroxypropylmethylcellulose (HPMC), were measured and compared to properties of similar yarns sized with the conventional size polyvinyl alcohol (PVA) unless stated otherwise. Polyvinyl alcohol was selected as the standard for comparison since it is generally recognized as the most universal size available. However, size formulations based on PVA are often modified with less expensive size materials. Yarns sized with PVA typically have a very high tensile strength and abrasion resistance, but the level of properties obtained with less expensive size materials is usually adequate for most weaving situations.

EXPERIMENTAL PROCEDURES

Application

Two slashers were used. One was a laboratory scale slasher which produced a sized warp one inch (1 in. 2.54 cm) wide (Figure 2).

The supply package was a beam 4 3/8 inches (11.1cm) wide. The yarn passed under a single immersion roll in an indirectly heated size box and was squeezed between a bottom brass roll and a top viton roll (55 durometer Shore A, cold). The squeeze pressure was regulated by spring loading each end of the top squeeze roll. Hot air was circulated through the drying chamber to remove the solvent or water from the warp. All warps consisted of one hundred twenty (120) ends. Prior to take-up on the loom beam, the yarn was split by a single lease rod and passed through an expansion comb with three (3) ends per dent. Tension on the warp was controlled via a spring loaded compensator roll which activated limit switches to increase or decrease the take-up speed as required. The yarn speed at the size box squeeze rolls was constant.

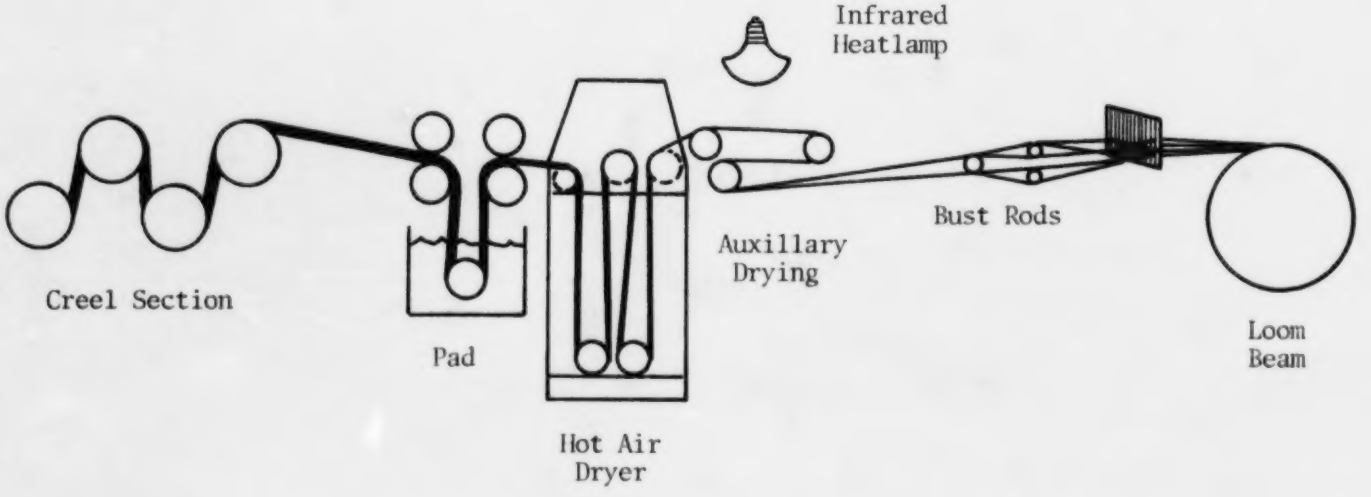

FIGURE 2. LABORATORY SLASHER

The second applicator was a Callaway Slasher. Yarns were pulled from a cone creel through the size mix at controlled width. The yarns were then dried by convection and/or conduction on electrically heated cylinders. Tension on the yarn was controlled by a weighted compensator roll. Take-up beam rpm was automatically decreased to compensate for beam diameter increase as the yarn was wound.

Size Add-on Determination

Size add-on was determined by weight loss of a sample upon washing. Size add-on and content were calculated by accepted procedures.[2]

Abrasion Resistance

The resistance of yarns to abrasive forces was measured using a Zweigle yarn abrasion tester. Twenty (20) specimens of yarn under predetermined tension (20 or 30 grams per yarn) were simultaneously rubbed by a standard 400 or 800 grit abrasive paper. The number of strokes required to cause each yarn to break was recorded.

Stress-Strain

Breaking tenacity and elongation of the yarns was measured on the Instron Tester using 10 inch (25.4 cm) gauge length and 2 inches/min (5.08 cm/min) crosshead speed.

Penetration into Yarn

Sized and unsized yarns were examined using a Scanning Electron Microscope and a light microscope to assess the uniformity of the coating of the size material on the yarn and to determine the degree of penetration of the size material into the yarn.

RESULTS AND DISCUSSION

Abrasion Resistance

The results of abrasion tests on the polyester/cotton blend yarns sized with HPC and PVA are shown in Table 4. Yarns sized with PVA at 9.5% size add-on averaged 175 abrasive cycles to break. Weaving tests established that this level of abrasion resistance was very adequate for good weaving performance with these yarns. The higher PVA add-on level (17.1%) improved the abrasion resistance to an average of 407 cycles. However, this level of abrasion resistance is not required for good weaving, and yarns containing over 15% PVA would be difficult to split at the slasher lease rods.

Experiments performed early in this project revealed that a minimum of approximately 10% add-on of HPC would be required for the 50/50 polyester/cotton yarns to be woven. Furthermore, solutions of HPC at concentrations

TABLE 4. ABRASION RESISTANCE[4] OF 36'S, 50/50 POLYESTER/COTTON YARNS SIZED WITH HPC AND PVA

Size Formula	Add-on (%)	Abrasion Resistance Cycles	Standard Error
Unsized	0	74	3.6
PVA[1]	9.5	175	13.6
PVA	17.1	407	36.7
HPC[2]	11.3	146	7.7
HPC[3] (reclaimed)	11.9	121	3.9
HPC/PVA 80/20 blend	7.2	124	6.2
HPC/PVA 80/20 blend	10.8	194	8.4
HPC/PVA 90/10 blend	10.9	189	10.0
HPC/PVA 95/5 blend	10.4	219	9.7

[1]Elvanol T-66. 5% Griffwax based on the weight of the PVA was added.

[2]Hercules, Inc. Klucel J

[3]Formulation contained 1% Carbowax 20M (Union Carbide) based on the weight of the Klucel.

[4]Using 20g tension and 400 grit paper

greater than 10% by weight are too viscous to handle easily in the laboratory slashing equipment. Therefore, add-on levels of 10% to 12% were as high as we could obtain in the laboratory. The abrasion resistance of 50/50 polyester/cotton yarns sized with HPC at 11.3% add-on averaged 146 cycles. Using reclaimed HPC the abrasion resistance at 11.9% add-on averaged 121 cycles. The reclaimed HPC formulation contained 10% on weight of the HPC of Carbowax 20M. The presence of carbowax 20M caused the strength of HPC films to be lower and probably accounts for the lower abrasion resistance of the yarns sized with reclaimed HPC as compared to yarns sized with virgin HPC. Yarns sized with either virgin HPC or reclaimed HPC were successfully woven during this project using a 24-inch Draper E-Model loom.

The abrasion resistance of 50/50 polyester/cotton yarns sized with mixtures of HPC and PVA is also shown in Table 4. A size consisting of 95 parts of HPC with 5 parts of PVA gave yarns having much better abrasion resistance than yarns sized with HPC alone. Increasing the amount of PVA to 10, 15 and 20 parts did not give further improvement in abrasion resistance. Apparently, a small amount of PVA in the size formulation improves the adhesion of the size to the fibers giving them better resistance to abrasion.

The abrasion resistance of 50/50 polyester/cotton yarns sized with mixtures of HPC with various other size materials is shown in Table 5. As previously stated, the presence of PVA in the formulation caused a marked improvement in abrasion resistance of the yarn. The presence in the size formulation of CMS, a low degree of substitution carboxymethylated starch, or Eastman WD, an aliphatic polyester-based size, caused a small to moderate improvement in abrasion resistance of the sized yarn compared to yarn sized with HPC alone at equivalent size add-on. The presence of HEC, a water soluble hydroxyethylated starch gave lower wet pickup than was anticipated resulting in a low size add-on and poor abrasion resistance. Because the viscosity of the HEC/HPC solution was higher than was desirable, additional work was not done with the formulation. The significance of the results of this work with size material mixtures is that the size material costs may be lowered by adding in a lower cost material or certain yarn physical properties may be improved by addition of small amount of binders such as PVA or liquid polyester sizes.

The results of abrasion tests performed on yarns sized with formulations based on HPMC, hydroxypropylmethylcellulose, are shown in Table 6. The HPMC used in this work, an experimental polymer, was recoverable at a level of about 40-60%. The viscosity of the recovered HPMC was much higher than the viscosity of the virgin HPMC at the same total solids concentration indicating that the portion of HPMC lost in the recovery process was lower in molecular weight than was the recovered HPMC. The abrasion test results characterize yarns sized with virgin HPMC. The HPMC gave yarns having somewhat lower abrasion resistance than yarns sized with HPC at similar size add-on levels. The presence of PVA or CMS in size formulations based on HPMC did not cause significant improvement in yarn abrasion resistance. Yarns containing less than 12% HPMC were successfully woven in this project on a 24-inch Draper E-Model loom.

TABLE 5. ABRASION RESISTANCE[6] OF 36'S 50/50 POLYESTER/COTTON YARNS SIZED WITH BLENDS OF HPC WITH SELECTED SIZE MATERIALS

Size Formula	Add-on (%)	Abrasion Resistance Cycles	Standard Error
Unsized	0	74	3.6
HPC	11.3	146	7.7
HPC/PVA[2] 80/20 Blend	10.8	194	8.4
HPC/CMS[3] 80/20 Blend	10.4	140	6.4
HPC/HEC[4] 80/20 Blend	6.9	104	3.0
HPC/Polyester[5] 80/20 Blend	10.1	170	6.4

[1] Hercules, Inc. Klucel J.

[2] DuPont, Elvanol T-66.

[3] Penick and Ford, Ltd. Astragum 3020.

[4] Penick and Ford, Ltd. Penford Gum 380.

[5] Eastman Chemical Products, WD size.

[6] Using 20g tension weights and 400 grit emery paper.

TABLE 6. ABRASION RESISTANCE[4] OF 36'S, 50/50 POLYESTER/COTTON YARNS SIZED WITH FORMULATIONS CONTAINING HPMC[1]

Size Formula	Add-on (%)	Abrasion Resistance Cycles	Standard Error
Unsized	0	74	3.6
HPMC[1]	10.9	113	4.9
HPMC	16.1	122	6.5
HPMC/PVA[2] 80/20 Blend	16.1	123	6.4
HPMC/CMS[3] 80/20 Blend	18.1	120	5.8

[1]Dow Chemical Company, Methocel J-15 experimental polymer

[2]DuPont, Elvanol T-66

[3]Penick and Ford, Ltd. Astragum 3020

[4]Using 20g tension weights and 400 grit paper

TABLE 7. ABRASION RESISTANCE[3] OF 34'S 100% COTTON YARNS SIZED WITH HYDROXYPROPYLCELLULOSE (HPC)

Formulation	Size Add-on (%)	Abrasion (cycles)	Standard Error
Unsized	0	73	3.5
HPC[1] (virgin)	14.0	123	4.9
HPC[1] (virgin)	18.7	183	10.9
HPC[2] (reclaimed)	15.2	104	6.5

[1]Hercules, Inc. Klucel J

[2]Contained 1% on weight of HPC of Carbowax 20M

[3]Using 20g tension weights and 400 grit paper

The results of abrasion tests on 100% cotton yarns sized with HPC are shown in Table 7. Either virgin HPC or reclaimed HPC improved the abrasion resistance of 100% cotton yarns. Somewhat higher abrasion resistance was achieved with virgin HPC than with reclaimed HPC at similar size add-on levels. The lower abrasion resistance of yarns sized with reclaimed HPC is the result of contamination of the HPC by natural impurities extracted from the fabric during desizing and by the presence of Carbowax 20M which was added for the purpose of producing a harder HPC film. Since the viscosity of the reclaimed HPC solution was as high as that of the virgin HPC solutions, the use and reclamation of HPC apparently does not cause molecular degradation of the HPC.

Stress-Strain

The breaking tenacity of the yarns was increased by addition of the size materials as indicated in Table 8. The effect of HPC on the breaking strength and elongation of the 50/50 polyester/cotton yarns was about the same as the effect of PVA. The strength of the yarns increased by about 15% as a result of sizing with either PVA or HPC. The elongation of the sized yarns was about 20 to 30% lower than that of the unsized yarns. These properties are typical for yarns adequately sized for weaving.

Results[3] of tests of tenacity of 22's cotton and 22's, 50/50 polyester/cotton yarns are shown in Tables 9 and 10. The effect of size on the polyester/cotton 22's was similar to the effect on the polyester/cotton 36's. A moderate increase in yarn strength and a moderate decrease in elongation at the break occurred in the sized yarn compared to the unsized yarn regardless of whether the size was PVA or HPC.

The tenacity of the 100% cotton yarns was increased dramatically by application of either PVA or HPC. The amount of increase in tenacity was influenced more by size add-on level than by the nature of the size. The loss in elongation experienced in slashing was not significantly different for formulations based on PVA and HPC and was not a function of size add-on level. Therefore, the yarn elongation is influenced more by slashing technique than by the nature of the size applied. The data obtained in this project on yarn strength and elongation are typical for yarns adequately sized for weaving.

Penetration into Yarn

A red tracer dye was added to an HPC size formulation and a PVA size formulation applied to 50/50 polyester/cotton and 100% cotton yarns. Microscopic examination of cross sections of these yarns did not reveal any difference in the degree of penetration of these two sizes even though the viscosity of the formulation based on HPC was much higher than that of the formulation based on PVA. With both sizes the penetration was 1/3 to 1/2 the distance from surface to center of the yarn. The good penetration of HPC into the yarn can be rationalized since HPC is very surface active in water. The surface tension of solutions of HPC is low which allows the solution to quickly wet the fiber surfaces so that penetration of size solution into the yarn bundle is rapid.

TABLE 8. PROPERTIES OF 36/1, 50/50 POLYESTER/COTTON YARNS SIZED WITH POLYVINYL ALCOHOL (PVA)[1] OR HYDROXYPROPYLCELLULOSE (HPC)[2].

Sample Description	Size Concentration In Formulation (%)	Break Factor	Elongation (%)
Unsized Control	--	432	13.4
PVA with lubricant	10	500	10.7
HPC	10	501	11.0

[1]DuPont, Elvanol T-25

[2]Hercules, Klucel J.

TABLE 9. BREAKING STRENGTH[3] OF 22's, 50/50 POLYESTER/COTTON AND 22's, 100% COTTON YARNS SIZED WITH HYDROXYPROPYLCELLULOSE[1] (HPC) OR POLYVINYL ALCOHOL[2] (PVA).

Size, Yarn	Size Add-on (%)	Mean Break Factor (oz. x counts)	Standard Error	% Increase Compared to Unsized Yarn
Polyester/Cotton/Unsized	0	363	7	-
Polyester/Cotton (PVA)	7.0	384	7	6
Polyester/Cotton (HPC)	13.8	426	14	17
100% Cotton (unsized)	0	268	7	-
100% Cotton (PVA)	4.2	320	7	19
100% Cotton (PVA)	10.8	352	11	31
100% Cotton (HPC)	6.1	313	11	17
100% Cotton (HPC)	10.0	352	11	31
100% Cotton (HPC)	15.0	359	7	34

[1]Hercules, Inc. Klucel J

[2]DuPont, Elvanol T-25. The formulation contained 5% Griffwax based on the weight of the PVA.

[3]Data from Reference #3.

TABLE 10. ELONGATION[3] of 22's, 50/50 POLYESTER/COTTON AND 22's, 100% COTTON YARNS SIZED WITH HYDROXYPROPYLCELLULOSE[1] (HPC) OR POLYVINYL ALCOHOL[2] (PVA).

Size, Yarn	Size Add-on (%)	Elongation at Break (%)	Standard Error	% Decrease Compared to Unsized Yarn
Polyester/Cotton (unsized)	0	11.?	0.1	-
Polyester/Cotton (PVA)	7.0	7.9	0.2	30
Polyester/Cotton HPC	13.8	8.1	0.3	28
100% Cotton (unsized)	0	7.0	0.1	-
100% Cotton (PVA)	4.2	5.4	0.2	24
100% Cotton (PVA)	10.8	5.5	0.2	21
100% Cotton (HPC)	6.1	-	-	-
100% Cotton (HPC)	10.0	5.5	0.1	26
100% Cotton (HPC)	15.0	5.2	0.1	26

[1]Hercules, Inc. Klucel J

[2]DuPont, Elvanol T-25. The formulation contained 5% Griffwax based on the weight of the PVA.

[3]Data from Reference #3.

SECTION 6

WEAVING OF HPC SIZED YARNS

PROCEDURES

Yarn Preparation

Yarns for weaving have been prepared in three different ways during this investigation. First, 120 individual yarns were sized on a small sample machine. These sized yarns were then back wound in equal sections onto ten beams which were mounted on a common shaft giving 1200 yarns for weaving. The second sizing procedure involved the use of a single beam containing 1440 yarns sized on a 20-inch wide sample slasher; and finally, 50-inch beams with 4404 yarns were sized in a textile plant on a full width slasher.

Weaving

Weaving trials have been conducted in the fabrics lab of the Department of Textile Engineering at Auburn and in the weaving operation of a nearby textile plant. Two looms were used in the fabrics lab trials: a 24-inch, Draper E-Model running at 175 weaving cycles per minute and a 50-inch, Draper X-3 Model running at 215 weaving cycles per minute. A 50-inch, Draper, X-3 Model loom running at 215 cycles per minute was used in the plant trials.

Early weaving trials at Auburn were conducted in uncontrolled atmospheric conditions; but because humitiy seemed to affect the weaving performance of HPC sized yarns, the test looms were isolated in a room where ambient conitions could be controlled and monitored. In the plant trials and most of the later trials at Auburn, room conditions were held constant at 70 degrees Fahrenheit and 70 percent relative humidity.

The determination of yarn performance during weaving was based on subjective observation and the collection of data on warp yarn breaks. The fabric density was held at approximately 90 warp yarns per inch and 80 weft or filling yarns per inch in all but a few trials. These few trials were run with fewer filling yarns per inch in order to move the warp yarn through the loom at a faster rate. The standard fabric organization is presented in Table 11.

RESULTS AND DISCUSSION

Early weaving trials conducted in uncontrolled atmospheric conditions in-

TABLE 11. FABRIC ORGANIZATION FOR WEAVING PERFORMANCE TESTS OF YARNS SIZED WITH EXPERIMENTAL POLYMERS

FABRIC - Blend Sheeting
WEAVE - Plain on 4 harnesses, drawn straight
REED GAUGE - 44.4 spaces per inch
TOTAL ENDS - 4404 drawn 2 per reed space
REED SPREAD - 49.32 inches
WARP YARNS PER INCH - Approximately 90
FILLING YARNS PER INCH - Approximately 80
SELVAGE - 40 yarns drawn 4 per reed space and 2 per heddle eye
YARN COUNT - 36.00 warp and filling
FIBER CONTENT - 50% cotton/50% polyester
YARN TWIST - 3.95 t.m.
FABRIC WEIGHT - 0.3153 lbs/yard
WARP WEIGHT - 0.1622 lbs/yard
FILLING WEIGHT - 0.1310 lbs/yard
SIZE WEIGHT - 0.0221 lbs/yard based on 12% add-on

dicated that high levels of relative humidity contributed to the generation of fuzz balls during weaving of HPC sized yarns. During the best of these early weaving trials a section warp containing HPC and PVA sized yarns ran for 6.8 hours with 7 yarn breaks occurring within the HPC sized portion while 2 yarn breaks occurred in the PVA sized portion.

Subjective evaluation of the first 20-inch wide warp sized with HPC supported the earlier observation that relative humidity was a critical factor to the weavability of HPC warps. Temperature and humidity controls were installed in the weaving lab at Auburn in an effort to determine the effect of these factors. Attempts to weave at levels of relative humidity above 70% failed on warps sized with HPC due to excessive fuzz-balling. At levels below 70% down to a level of 40% HPC sized warps ran well. As the level of relative humidity was decreased below 70% there appeared to be a corresponding increase in the amount of shed from warps sized with HPC and difficulties were again encountered in weavability below 40% relative humidity. All of the early weaving trials were done on the 24-inch, Draper E-Model loom with 175 filling insertions per minute.

Based upon these early trial results, a size solution containing 8.5% by weight of HPC was prepared for use in a mill trial. The size solution was diluted to about 7.5% solids content and a warp was produced with about 12% size content on the yarn. The warp contained slack yarns in the extreme outside edges, especially on the left-hand size of the warp. This left-hand side at the slasher later produced poor weaving results on the right-hand side of the loom. The slack yarns were the result of adjustments that were made at the slasher to overcome excessive size pick-up by the yarn. As can be seen in Table 12, the normal running conditions in the mill at the time of this trial were at a performance level of over 96 percent efficiency. The HPC test warp performed at just over 64 percent efficiency. There were three direct causes for the loss in efficiency on the HPC test warp. First, the weavers assigned to the job were not as attentive to the HPC loom (7.2 min.) after a stop as they were to other looms (4.1 min.). Second, the right-hand selvage (slack side from slashing) accounted for a disproportionate number of breaks as presented in Table 13. The slack right-hand selvage yarns failed in the loom over 4 times more often than did the left-hand selvage yarns. The third reason for efficiency loss on the HPC loom was a build-up of size material on the shuttle.

Buildup of HPC size occurred mostly on the top of the front wall of the shuttle, but generally, all exposed surfaces of the shuttles as well as other loom surfaces in the flight path of the shuttle were affected by the build-up. Occasionally a strip of size would be ripped off the shuttle and become woven into the fabric creating a defect that did not later wash out. Since this phenomenon had not been observed previously, the HPC warp from the mill trial was moved to the lab at Auburn for further evaluation.

The weaving difficulties which occurred at the mill also occurred in the trials at Auburn. The buildup of the material on the shuttle was caused by the soft, tacky nature of the HPC. The tackiness of the HPC can be reduced by lowering the relative humidity of the weaving area. Weaving tests were

TABLE 12 . LOOM EFFICIENCY FOR HPC SIZED WARP DURING FIRST MILL TRIAL*

	HPC WARP	NORMAL MILL DATA
Loom % Efficiency	64.2	96.3
Average Lost Minutes/Stop	7.2	4.1

*Test warp contained 4404 yarns woven through a 44.4 gauge reed with 2 ends drawn in each space weaving at 215 filling insertions per minute on a Draper X-3 Model loom.

TABLE 13 . LOCATION OF WARP YARN BREAKS IN HPC SIZED WARP DURING FIRST MILL TRIAL*

Yards of Fabric Run	340
Total Warp Yarn Breaks	372
Breaks within 3 inches of either Selvage	320
Breaks in Left-Hand Selvage	55
Breaks in Right-Hand Selvage	265

*Test warp contained 4404 yarns woven through a 44.4 gauge reed with 2 ends drawn in each space weaving at 215 filling insertions per minute on a Draper X-3 Model loom.

performed at several relative humidity levels between 85% and 40%. Weaving performance was extremely poor at 85%, but improved as the humidity was lowered. The reason size buildup had not surfaced in previous trials is felt to be related to the differences between test looms. The test run on smaller sample warps in the Draper E-Model loom did not produce size buildup to a disruptive degree. The opposite is true of full sample warps on Draper X-3 Model looms where all tests did exhibit the buildup of size to the point of interference with weaving performance.

A second warp was sized in the mill using a 90/10 mixture of hydroxypropylcellulose and plolyvinyl alcohol. Size add-on to this yarn was 17.0%. The yarn was difficult to split at the slasher lease rods; and as a result, two warps with several lost and several crossed ends were obtained. The in-plant results of this trial are presented in Table 14 where it is shown that the normal level of weaving performance during the test period was 93.7% efficiency. The mill's looms experienced 6.9 average stops per 8 hours while the two test looms ran at levels of 52.5 and 47 stops per 8 hours. Weavers were instructed to be attentive to the test looms to avoid a repeat of the first mill run. As seen in Table 14 the weavers were slightly more attentive to the test looms than to the rest of their job with an average lost minutes of 4.04 per stop for regular looms compared to 3.75 and 3.86 minutes for the test looms. The location of warp yarn breaks during this mill trial are shown in Table 15. The difference that existed between the left-hand and right-hand selvages during the first mill trial did not occur in this test. The buildup of size material on the shuttle did reoccur in this trial and was judged to be the primary cause of poor weaving performance. The presence of lost ends and crossed ends contributed to the efficiency loss as well.

One of the 90/10 HPC/PVA test warps was moved to Auburn for further evaluation. Weaving tests were performed with this warp at 70% relative humidity and at 50% relative humidity. The tendency of the size to accumulate on the shuttle was much lower at 50% RH than at 70% RH; however, the accumulation was not completely eliminated. The accumulated size material was less tacky at 50% RH than at 70% RH and was less tenaceously held by the shuttle. Therefore, the accumulated size material was periodically stripped off of the shuttle by the selvage yarns as the shuttle entered the shed. At 50% RH the warp was run for 2 hours before any selvage yarn breaks were caused by the accumulated size on the shuttle. At 70% RH this accumulation of size material occurred gradually and began to interfere with the entry of the shuttle into the shed after about 4000 filling insertions (or about 20 minutes). Yarns near the selvages of the fabric were plucked by the accumulated size as the shuttle entered the shed causing frequent yarn breaks to occur in both selvages, as shown in Table 16.

The number of picks per inch of fabric was reduced from 80 to 40 in order to determine if the size accumulation on the shuttle was related to repeated contact of the shuttle with the same yarns. In this way the warp yarns were subjected to fewer abrasive cycles as they passed through the weaving elements of the loom. As shown in Table 16, this more lightly constructed fabric was produced with few yarn breaks even though the buildup of material on the shuttle still occurred at about the same rate as it had in previous trials.

TABLE 14 . LOOM EFFICIENCY FOR 90/10 HPC/PVA SIZED WARPS DURING SECOND MILL TRIAL*

	Test Warp #1	Test Warp #2	Normal Mill Data
Loom % Efficiency	58.6	61.9	93.7
Average Stops/8 Hours	52.5	47.0	6.9
Average Lost Minutes/Stop	3.75	3.86	4.04

*Test warp contained 4404 yarns woven through a 44.4 gauge reed with 2 ends drawn in each space weaving at 215 filling insertions per minute on a Draper X-3 Model loom.

TABLE 15 . LOCATION OF WARP YARN BREAKS IN 90/10 HPC/PVA SIZED WARPS DURING SECOND MILL TRIAL*

	Test Warp #1	Test Warp #2
Yards of Fabric Run	34	127
Total Warp Yarn Breaks	50	151
Breaks within 3 inches of either Selvage	35	101
Breaks in Left-Hand Selvage	15	45
Breaks in Right-Hand Selvage	20	56

When the shuttle was cleaned thoroughly every 20 minutes no weaving problems were encountered except those related to the lost and cross ends mentioned previously. Adding a device to wipe the size from the shuttle as each pick was inserted had no effect on the accumulation. This and the observation that significant accumulation of the size on the shuttle occurs only on the top-front edge of the shuttle leads to the conclusion that the size transfers directly to the shuttle upon its contact with the yarn.

A test was performed using a wooden shuttle instead of the phenol/formaldehyde laminated cotton fabric shuttle used in previous weaving tests. The purpose of this test was to learn if the HPC would accumulate on wood as it had on the composite material. The size material did accumulate on the wooden shuttle at about the same rate that it had accumulated on the composite shuttle.

Table 16. LOCATION OF WARP YARN BREAKS IN 90/10 HPC/PVA SIZED WARP DURING TRIALS AT AUBURN*

	80 Picks per inch	40 Picks per inch
Yards of Fabric Run	12.5	2.0
Total Warp Yarn Breaks	114	3
Breaks within 3 inches of either Selvage	111	1
Breaks in Left-Hand Selvage	57	1
Breaks in Right-Hand Selvage	54	0

* Test warp contained 4404 yarns woven through a 44.4 gauge reed with 2 ends drawn in each space weaving at 215 filling insertions per minute on a Draper X-3 Model loom.

The effect of a silicone spray on size buildup on the shuttle was tested. The silicone spray lubricant decreased the rate of buildup of size on the shuttle but was effective for only a few thousand pick insertions. Apparently, the lubricant wears off in a relatively short weaving time.

A different polymer, hydroxypropylmethylcellulose (HPMC), supplied by Dow Chemical Company was also tested for weaving performance. The first trial on HPMC was run at Auburn in between the mill trials on HPC. The results of this test which was designed to check response of the HPMC to humidity changes are presented in Table 17. It can be generally stated that

both the percent shed falling off the HPMC sized yarns and the weaving performance of the yarns was better at the higher levels of %RH in this trial.

TABLE 17. DATA FROM WEAVING* of HPMC SIZED WARP

F° - %RH	% SHED	YDS WOVEN/WARP BREAK
75° - 70%	-	7.33
70° - 70%	0.34	6.72
70° - 60%	0.44	4.94
70° - 50%	0.63	5.63
70° - 40%	-	1.76

* Test warp contained 1200 yarns drawn through a 43.0 gauge reed with 2 ends drawn in each space weaving at 175 filling insertions per minute on a Draper E-Model loom.

One other weaving trial was conducted involving HPMC during the time period between plant trials on HPC and the results of this run are shown in Table 18. This section warp contained yarns sized with recycled HPC alone and with wax (2% Carbowax) and yarns sized with HPMC that had been purified by thermal precipitation. Only two segments of the warp experienced difficulties. The HPC sized section contained one selvage end that broke repeatedly during the second 10-yard segment. The HPC with wax experienced a similar problem at the third 10-yard segment. Otherwise, all of the sections ran well.

Dow Chemical supplied a modified version of its hydroxypropylmethylcellulose (HPMC) which according to Dow is stiffer and less hygroscopic than the HPC and other HPMC products previously used. Sizing trials were run at Auburn using a formulation containing a 90/10 mixture of this HPMC with PVA. Only enough of the experimental HPMC was available to size a 4 1/2 inch strip of yarn for each side of the Draper X-3 loom which was occupied by the warp sized with the 90/10 HPC/PVA formulation. The first 450 yarns on each side of the HPC warp were replaced by the HPMC sized yarns. The remaining 3504 yarns still contained HPC. Size material accumulated on the shuttle in the weaving trials which were subsequently performed; and, it could not be determined whether the accumulation was mostly HPC or HPMC. The buildup did not occur as rapidly as in previous tests. The HPMC sized portions of the warp were moved to the Draper E-Model loom where sixteen hours

of running revealed no significant buildup on the shuttle or other loom surfaces. The polymer available was insufficient to size a full-width warp so the question as the whether HPMC would buildup in the faster Draper X-3 loom could not be answered.

TABLE 18. NUMBER OF WARP YARN BREAKS OCCURRING DURING THE WEAVING* OF HPC AND HPMC SIZED YARNS

CUMULATIVE YARDS WOVEN	HPC RECYCLED	HPC RECYCLED & WAX ADDED	HPMC RECYCLED
10	0	0	0
20	10**	0	0
30	0	6***	2
40	0	1	0
50	1	0	0
60	1	1	0
TOTAL	12	8	2

* Test warps contained 360 yarns in each section drawn through a 43.0 gauge reed with 2 ends drawn in each space weaving at 175 filling insertions per minute on a Draper E-Model loom.

** 9 of 10 breaks occurred on the same selvage end.

*** 4 of 6 breaks occurred on the same end.

SECTION 7

THERMAL PRECIPITATION AND SEPARATION OF HPC FROM WATER

INTRODUCTION

Hydroxypropylcellulose (HPC) is soluble in water at temperatures lower than 43°C (109°F) and can be removed from a woven fabric with water at temperatures below 43°C (109°F). The tendency of HPC to dissolve in water improves as the temperature is lowered, but HPC will readily dissolve in water at temperatures as high as 40°C (107°F). Data regarding the rate of removal of HPC and PVA from a woven fabric is shown in Table 19. Fabric samples weighing approximately three (3) grams were placed in 100 ml. of distilled water at the specified temperatures (95°C for PVA and 25°C for HPC). The specimens were gently agitated and the weight (mainly size) loss with time was determined. Removal of most of the PVA or HPC was accomplished in 2.0 to 3.0 minutes. These figures indicate that HPC can be removed from the fabric at about the same rate using water at 25°C as PVA can be removed using water at 95°C. This information is significant for two reasons. First, the production and washing efficiency of the desize washer depends on the rate of removal of the size material from the fabric. Secondly, very high washing efficiency is required for an economic size reclamation system since the size not removed from the fabric in desizing is lost and must be replaced.

When water containing dissolved HPC is heated to a temperature above 43°C the HPC separates abruptly from water forming a white, milky suspension. The precipitation is accompanies by an abrupt decrease in viscosity (Figure 3) which is indicative of virtually complete insolubilization of the HPC polymer chains. The thermally precipitated HPC particles are small and apparently carry a surface charge which causes the suspension of HPC in water to behave colloidally.

SEPARATION OF HPC FROM WATER BY FILTRATION AND CENTRIFUGATION

The heart of the reclamation system for HPC is the separation of the thermally precipitated HPC particles from most of the water to obtain a size solution sufficiently concentrated to reuse in slashing. Separation of the precipitated particles by filtration and centrifugation each appeared feasible in laboratory experiments. Filtration experiments were performed using a heated Buchner funnel and Watman No. 1 filter paper in a suction filtration

TABLE 19. RATE OF EXTRACTION OF HPC AND PVA FROM FABRIC.

Treatment Time (minutes)	Weight % Extracted from Fabric PVA[a]	HPC[b]
0.5	4.4	3.5
1.0	5.1	5.0
1.5	4.9	5.5
2.0	-	6.5
3.0	5.0	6.9
x[c]	5.8	6.9

[a] Test performed at 95°C (203°F) water temperature

[b] Test performed at 25°C (77°F) water temperature

[c] Samples were completely desized using three charges of clear water each for 3.0 minutes.

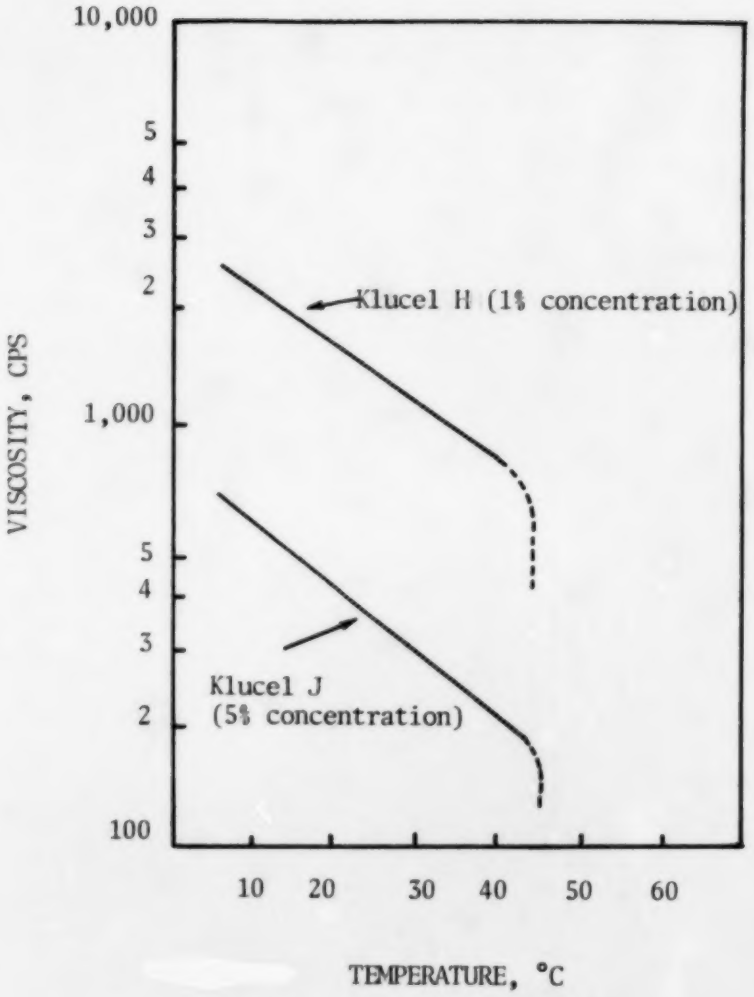

FIGURE 3. RELATIONSHIP BETWEEN VISCOSITY AND TEMPERATURE OF SOLUTIONS OF HPC (KLUCEL®).

device. About 90% recovery of a commercially available grade of HPC was obtained.* The HPC recovered by filtration formed a concentrated gel when allowed to cool. The concentration of the reclaimed HPC was about 40% by weight in water. The concentrated HPC could be diluted with water to make a solution of the concentration required for use in the sizing process.

Results with centrifugation were similar to those obtained with filtration. A 1% w/w solution of HPC in water, heated above the precipitation temperature, was placed in a laboratory centrifuge for thirty (30) seconds. Clear water was decanted off leaving the HPC in the bottom of the tube. Upon cooling a gel containing about 50% HPC by weight was obtained. This gel was diluted with water, and the HPC redissolved when stirred forming a solution of the concentration required for sizing. No HPC could be detected in the water which had been decanted off.

SEPARATION OF PRECIPITATED HPC FROM WATER BY SETTLING

Hydroxypropylcellulose (HPC) precipitates from desize water as a colloidal suspension upon heating. The precipitated particles of HPC do not settle well in pure water because the particles carry a negative surface charge, i.e. they have a negative Zeta Potential. The negative Zeta Potential causes the particles to repel each other and remain suspended.

Settling Assistants

Additives may be used to reduce the Zeta Potential allowing the precipitated HPC particles to coagulate into larger particles which will settle rapidly in an unagitated vessel. The effect of a number of chemicals on the propensity of precipitated HPC particles to settle in water was investigated. A number of substances which cause precipitated HPC particles to settle are listed in Table 20. It must be emphasized that these agents do not affect the solubility of HPC in water at the level that they are used to cause HPC to settle. Rather, the agents merely decrease the negative Zeta Potential and allow the already thermally precipitated HPC particles to coagulate and settle rapidly. If the water is allowed to cool, the HPC readily redissolves forming a smooth solution.

The efficiency of the coagulating effect of the agents listed in Table 20 increased with increasing concentration of the agent in all experiments performed in this work. As little as 0.001% by weight of aluminum sulfate dramatically increased the settling rate of the precipitated HPC from a water suspension containing 1% HPC by weight. About 0.005 to 0.01% by weight of aluminum sulfate gave rapid settling of precipitated HPC.

* Commercial grades of HPC contain impurities which are not recoverable in a thermal precipitation process. This means that a portion of the product will be lost after its initial use. However, the material lost is not all HPC and recovery levels in subsequent passes through the system are higher than in the initial recovery cycle.

When the concentration of HPC in the desize wastewater was much less than 1% by weight, a higher concentration of coagulating agent was required to cause rapid settling of HPC. For example, when the concentration of HPC in the desize washwater was 0.25 to 0.50% by weight, 0.02 to 0.03% by weight of aluminum sulfate was required for rapid settling to take place.

The coagulation of precipitated HPC particles by the agents listed in Table 20 depends on the nature of the cations in the substance. The ability of a substance to improve the propensity of precipitated HPC to settle depends on the valence of the cation in the substance used. For example, at equal ion concentrations Al^{+3} is more efficient than Ca^{+2} or Mg^{+2} which in turn are more efficient than Na^{+1} ions. In fact, the most efficient substance studied with regard to improving the settling rate of precipitated HPC was aluminum sulfate [$Al_2(SO_4)_3 \cdot 18\ H_2O$]. Figure 4 illustrates the settling rate of precipitated HPC containing 0.01% of a aluminum sulfate on the weight of the solution. The volume of the test solution was 450 ml., and it contained 4.5 grams of HPC. In 15 to 20 minutes virtually all of the precipitated HPC settled into the bottom 45 ml of the container, a 500 ml graduated cylinder. After the supernatant water was decanted or pipeted away, the precipitated HPC was allowed to cool so that it would redissolve, and its concentration was measured. Experiments of this type yielded solutions of reclaimed HPC containing between 8% and 11% by weight of HPC when aluminum sulfate was used to assist settling.

In summary, aluminum sulfate is an effective agent to facilitate the settling of thermally precipitated HPC in water. Aluminum sulfate was used in all of the recovery work on HPC removed from fabrics woven in this project. The presence of aluminum sulfate in the washwater had no detectable effect on the washing efficiency of the water.

Thermally precipitated hydroxypropylmethylcellulose (HPMC) did not require the presence of cations for rapid settling. The presence of Al^{+3} and other cations caused no detectable difference in the settling rate of HPMC particles.

Assay of Aluminum in Reclaimed HPC

Both the reclaimed HPC concentrate and the water separated from the HPC in the recovery system can be reused. After a typical recovery cycle approximately 10% of the desize washwater remains with the HPC concentrate which is reused in slashing. The remaining 90% of the washwater can be recycled to the desize washer. The concentration of impurities in the recycled water will increase each time the water is recycled. Therefore, it is necessary to periodically discharge some desize washwater and replace it with fresh water. Further, any Al^{+3} ions lost in the effluent would have to be replaced since efficient settling of HPC particles depends on the presence of a certain concentration of Al^{+3}. Therefore, it is important to know the distribution of Al^{+3} between the reclaimed concentrate and recycled water.

A potentiometric titration procedure and atomic absorpiton spectroscopy were used to measure the Al^{+3} concentration in the reclaimed concentrate and the recylced water. These results revealed that when 0.01% of aluminum sulfate on weight of the solution is used most of the Al^{+3} accom-

TABLE 20. EFFECT OF VARIOUS SUBSTANCES ON THE SETTLING RATE OF THERMALLY PRECIPITATED HPC IN WATER.

Substance	Magnitude of Effect	Concentration Required for Good Separation (% w/w in solution)
Sodium Chloride	Fair	0.05
Sodium borate	Poor	0.10
Ammonium chloride	Fair	0.05
Calcium chloride	Good	0.02
Magnesium chloride	Good	0.02
Aluminum chloride	Excellent	0.01
Various Cationic Polyelectrolytes	Good	-
Cationic Modified Guar Gum	Good	-

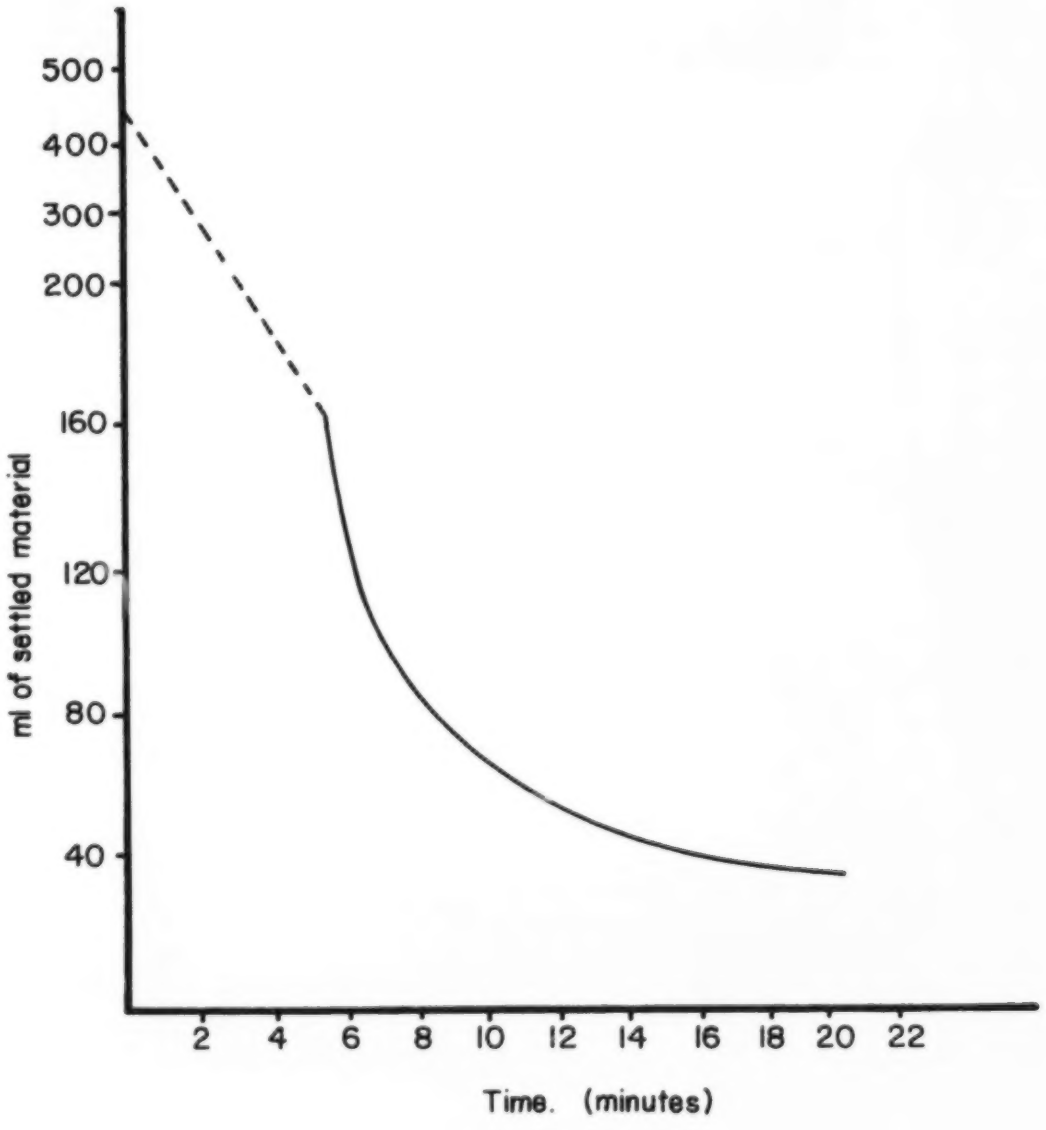

FIGURE 4. SETTLING RATE OF PRECIPITATED HPC AT 50°C IN WATER CONTAINING 0.01 PERCENT ALUMINUM SULFATE

panies the HPC in the concentrated solution and little Al^{+3} is present in the water returned to the desize washer. This means that little of the aluminum will be discharged with water which must be wasted from the system. If the amount of Al^{+3} present in the system is increased, a larger portion of the Al^{+3} ions is likely to remain in the water returned to the desize washer.

Several polyelectrolytes of the type commonly used to coagulate solids in water and wastewater treatment were tested for their propensity to cause precipitated HPC to settle in water. Polyelectrolytes of the anionic type were, as expected, ineffective as settling assistants for HPC.

Cationic polyelectrolytes proved to be very effective in causing thermally precipitated HPC to settle. Cat-Floc ^{R}, Cat-Floc T ^{R} and Cat-Floc T-1 ^{R}, strongly cation liquid polymers obtained from Calgon Corporation, all caused rapid settling of HPC particles. Approximately 0.01% of these agents on weight of the solution was required to promote rapid settling. WT-2575, WT-2635 and WT-2640, cationic polymers also supplied by Calgon Corporation, were less effective, than the Cat-Floc series. Two cationic flocculants, AP-30 and N-10 obtained from Dow Chemical Company were ineffective in promoting settling of HPC.

Cationic polyelectrolytes are no more effective than aluminum sulfate in causing thermally precipitated HPC to settle. Since the polyelectrolytes are more expensive than aluminum sulfate, there seems to be no advantage in their use in the thermal precipitation size reclamation process.

SETTLING SYSTEMS

Early experiments regarding settling of thermally precipitated HPC particles were done in 500 ml and 1000 ml graduated cylinders heated in a water bath. The HPC particles were allowed to settle and the supernatant water was decanted from the top leaving the HPC concentrated in the remaining water. To further demonstrate the applicability of the settling principle, several other experiments were done. A 30 liter sample of desize washwater containing 1.0% by weight of virgin HPC and 0.01% by weight of aluminum sulfate was heated to 55°C (131°F) in a jacketed stainless steel hemispherical tank. The HPC settled and after 20 minutes 28 liters of water was siphoned off. Approximately 2 liters of a 13% solution of HPC remained in the tank. This represents 85% recovery of the HPC originally present in the desize washwater.

A glass device for batchwise separation of HPC by settling was also fabricated. A schematic diagram of the device, which had a capacity of 750 ml, is shown in Figure 5. The desize washwater is heated to the precipitation temperature of HPC by warm water circulating in the jacket. The position of tube A which is used to remove the water from above the settled HPC is adjustable to the point of the interface between the settled HPC and the water. The HPC concentrate is removed after the settling stage through tube B.

Using this batchwise separation device a 1% solution of HPC, as obtained from the HPC supplier, can be separated in 15 minutes to give a 9.0% solution of HPC with 85% of the HPC originally present being in the concentrate. If the concentrated HPC solution is diluted to 1% concentration with fresh water and the separation repeated, a 9.0% solution of HPC can be obtained with more than 95% of the HPC recovered in the first separation being recovered in this separation. This means that the HPC as obtained from the manufacturer contains a portion which is not recoverable. This portion which is not recoverable has not been identified. Possibly, it is low molecular weight HPC which does not precipitate in warm water. The nonrecoverable portion may also be some other soluble impurity in the HPC.

Further concentrating of reclaimed HPC solutions can be accomplished by subjecting the 9.0% solution in the first separation to a second separation cycle in the batchwise separator. After settling for 15 minutes, a 9.0% solution can be concentrated to 25% HPC by weight. Higher solids concentration in the reclaimed size solution is advantageous if the reclaimed size must be transported from one location to another before reuse.

A device, shown schematically in Figure 6, to allow continuous separation of HPC from desize washwater was also fabricated. The principle of separation of HPC with this device also required settling of the precipitated HPC in water. The desize washwater is forced through tube A into the chamber which has a capacity of 750 ml. Reclaimed water flows from tube B and the layer of HPC concentrate which settles to the bottom of the container is removed at the required rate through tube C. At a flow rate into the separator of 25 ml per minute the recovery level was 89% of the HPC originally added to the water. As previously explained a large portion of the 11% of HPC not recovered is a soluble impurity in the HPC as received from the manufacturer. The concentration of HPC in the reclaimed HPC solution was 15.4% at the washwater flow rate of 25 ml/minute. At higher flow rate the recovery level and the concentration of the recovered solution are lower.

Reclamation of larger amounts of HPC from desize washwater resulting from desizing of fabrics produced during the weaving trials performed in this project was accomplished in a 20 liter glass bottle. Heat was applied by placing the bottle containing the desize washwater in a water bath. After settling had occurred the supernatant water was siphoned off.

These settling experiments demonstrated that HPC can be efficiently separated from desize washwater by either batchwise or continuous methods. No attempt was made to further refine the separation device since technology of solid/liquid separation based on settling is already available.

REUSE OF DESIZE WASHWATER

A major consideration in the function of a closed loop system such as size reclamation is the contamination of the system by impurities. Some impurities come from the nonrecoverable portion of the size as has been mentioned previously. The HPC which was used in this project contains some material (impurity) which does not precipitate when the washwater is heated.

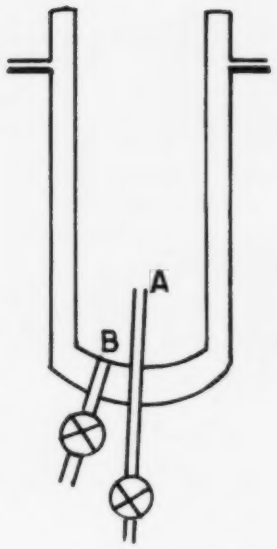

FIGURE 5. Size/Water Separator

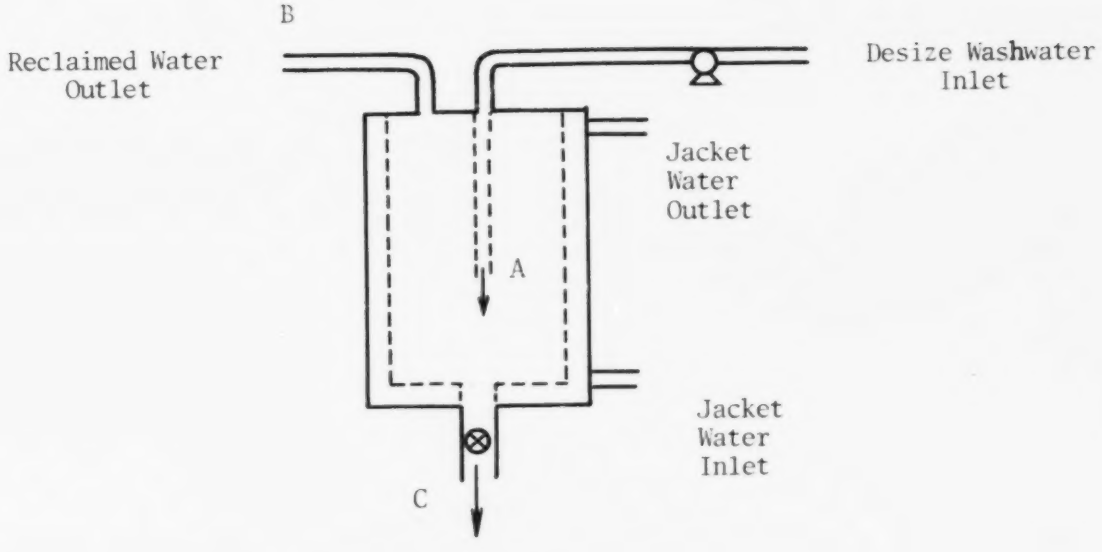

FIGURE 6. CONTINUOUS SEPARATOR

Therefore, this impurity remains dissolved in the water which is returned to the desize washer and in the water present in the size recycled to the slashing process. Other impurities such as dirt, lubricating oil, natural fats, natural waxes, spin finishes, etc. are removed from the fiber and fabric and become dissolved, emulsified or suspended in the recycled water and size. Each time the water and size is reused for desizing it becomes more discolored. Fortunately the discoloration of the size material when reused does not cause marked discoloration of the yarn to which it is applied. Further, any slight discoloration of the yarn is eliminated when the yarn is subsequently scoured and bleached. Therefore, the discoloration of the size due to buildup of impurities does not appear to present a problem.

The buildup of impurities in the recycled water and size material will be a problem if the impurities interfere with the washing efficiency of the reclaimed water or the properties of the reclaimed HPC. A series of experiments was performed to ascertain if water reclaimed from the thermal precipitation system can be reused for desizing. A sample of fabric containing HPC was desized in the amount of water required to give desize washwater containing about 1% by weight of HPC. The HPC was removed from the water by thermal precipitation and the recovered water was collected. The experiment was repeated for four (4) additional cycles using in each cycle the water which had been reclaimed from the previous cycle. The solids content of the desize washwater at each cycle is shown in Table 21. The solids content of the desize washwater both before and after the size was reclaimed increased with each cycle. This indicates that impurities are present and that the concentration of the impurity in the desize washwater increases with each cycle. The increase in residual solids content of the desize washwater averaged 0.24% on the weight of the desize washwater. A similar experiment was performed using yarns which contained no size. The data in Table 22 show that the buildup in residual solids in the washwater averaged 0.16% in this case. This means that about two-thirds (2/3) of the buildup results from impurities such as dirt, oil, grease, fats, waxes, salts, and sugars washed off of the fabric and fiber while the remaining one-third (1/3) of the buildup represents nonrecoverable impurities present in the HPC. Expressed as a percentage of the amount of HPC used, this nonrecoverable HPC represents a loss of 8.0% of all HPC purchased. However, as stated previously the percent recovery achievable is higher in subsequent cycles than in its initial use since less nonrecoverable material is present in the reclaimed HPC.

Another important observation from these experiments on reuse of desize washwater is that the water was still suitable for use in desizing after all five (5) cycles were completed. Since the concentration of impurities does increase each time the water is reused in desizing, a point might eventually be reached where the water would not effectively dissolve the size material. This point was not reached in five (5) desize cycles.

TABLE 21 . SOLIDS CONTENT OF RECYCLED DESIZE WASHWATER

Number of Cycles	% Solids Content of Washwater Before Size Recovery	% Solids Content of Washwater After Size Recovery	Increase in Residual Solids Content of Reclaimed Water (% on Weight of Reclaimed Water)
1	1.18	0.34	0.34
2	1.64	0.58	0.24
3	1.53	0.77	0.19
4	1.57	0.94	0.17
5	2.20	1.30	0.26
		Average	0.24

TABLE 22 SOLIDS CONTENT OF WATER USED TO WASH UNSIZED YARNS

Number of Cycles	% Solids Content of Desize Washwater	Increase in Residual Solids Content of Reclaimed Water (% on Weight of Reclaimed Water)
1	0.16	0.16
2	0.34	0.18
3	0.47	0.13
4	0.60	0.13
5	0.81	0.21
		Average = 0.16

SECTION 8

ECONOMIC EVALUATION OF SIZE RECLAMATION SYSTEMS

COST OF EQUIPMENT AND MATERIALS

The costs of reclamation of hydroxypropylcellulose (HPC) by thermal precipitation are shown in Table 23. These costs are based on the recovery system shown schematically in Figure 7 and the material balances shown in Figure 8. For purposes of comparison, two other costs are presented. These are the costs of reclamation of polyvinyl alcohol (PVA) by an ultrafiltration system and the desizing and wastewater treatment costs for conventional sizing without size reclamation. The ultrafiltration costs were derived from published data pertaining to the system of Abcor, Inc. The costs for conventional desizing without size reclamation were calculated based on typical procedures and materials used for polyester/cotton blend fabric production in the textile industry.

The cost figures for thermal precipitation and ultrafiltration pertain only to recovery system purchase, depreciation, operation, maintenance and wastewater treatment. Other sizing and desizing costs for the three systems should be essentially equal. The assumption is made that the size material, HPC or PVA, is reclaimed and reused at the same geographical location so that transportation of the reclaimed size is not necessary. Equivalent weaving performance of all sizes is also assumed.

Table 23 shows a summary of the costs analysis. The basis for the figures is a plant processing 600,000 pounds per week of a polyester/cotton blend material containing 6% size by weight. The desize washwater contains 1% by weight of size material so that the reclamation system must process about 3000 gallons of desize washwater per hour. Both the thermal precipitation and the ultrafiltration system concentrate the size to 8% solids by weight for return to the slasher.

As the Table shows, the annual operating cost for either an ultrafiltration system or a thermal precipitation reclamation system is less than for a conventional system, 18% and 14% respectively. The savings in the thermal precipitation recovery system result mainly from lower energy consumption and lower wastewater treatment costs while with ultrafiltration the savings is primarily from lower size material costs and lower wastewater treatment costs. The capital investment cost for an ultrafiltration system is more than 3 times that of a thermal precipitation system.

All three systems require an initial purchase of 25,000 pounds of size. Because the different size materials have different costs (approximately

TABLE 23. SUMMARY OF COSTS OF WARP SIZE RECLAMATION

	Ultrafiltration	Thermal Precipitation	Conventional
Fixed Capital Investment	$787,800	$212,590	--
Initial Size Investment	17,500	50,000	$6,250
Total Capital Investment	$805,300	$262,590	$6,250
Annual Operating Costs Including Depreciation[1]	$596,410	$625,350	$733,000
Return on Investment	17.1%	42.0%	-
Payout Time, years	3.74	2.04	-
Discounted Cash Flow Rate of Return	10.4%	33.4%	-

[1] Includes size replacement for size lost in processing, hot water for desizing and waste water treatment as well as other operating costs.

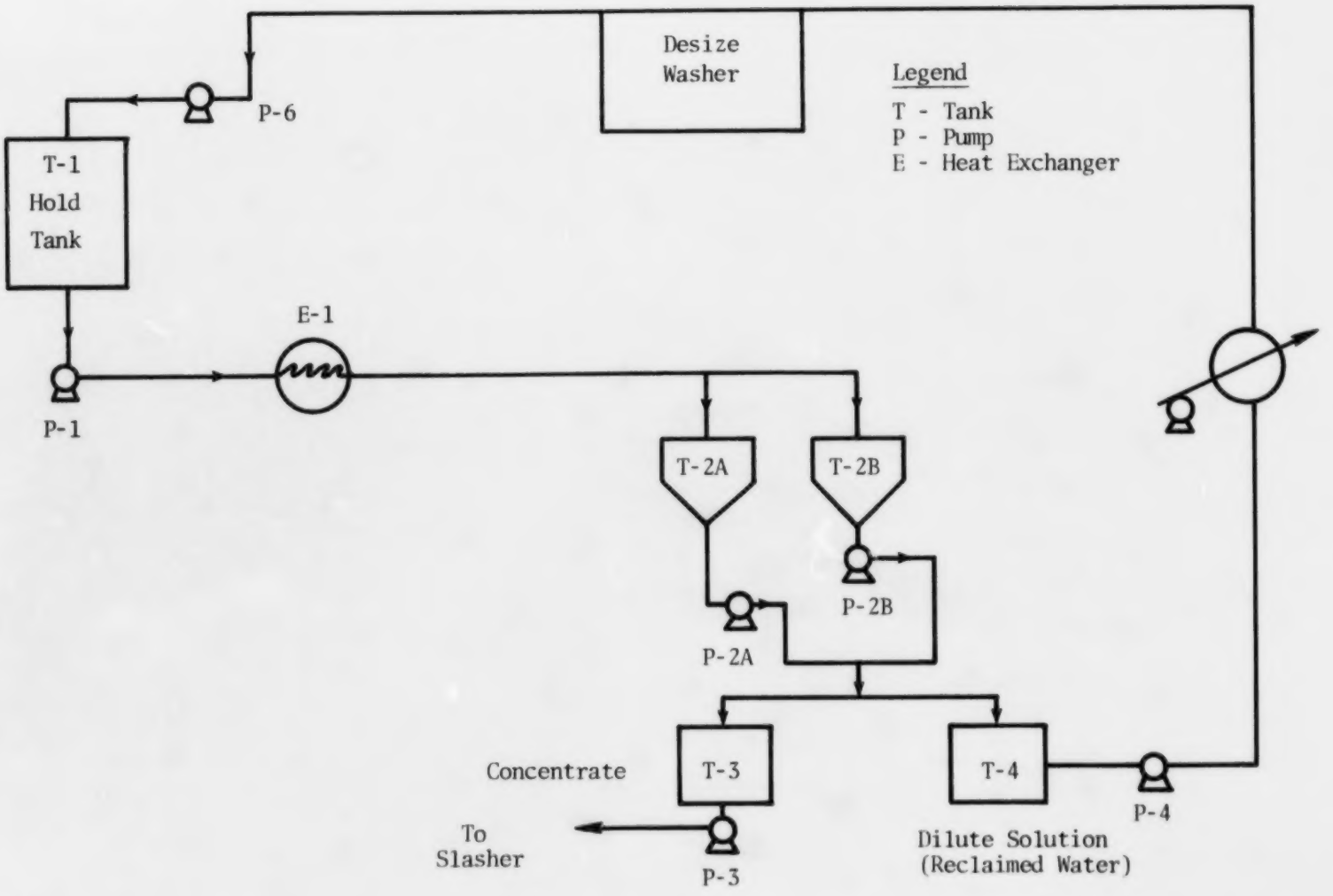

FIGURE 7. THERMAL PRECIPITATION RECLAMATION SYSTEM.

Item	Input (lbs./100 lbs. fabric)	Recovered (lbs./100 lbs. fabric)	Output (lbs./100 lbs. fabric)
HPC Size Material	6.0	5.16	0.84
Water	727.3	59.35 (recycle to slasher)	169.0 (by evaporation)
		399.2 (recycle to washer)	99.8 (as effluent)

Legend: H = HPC; W = Water; Y = Yarn; F = Fabric

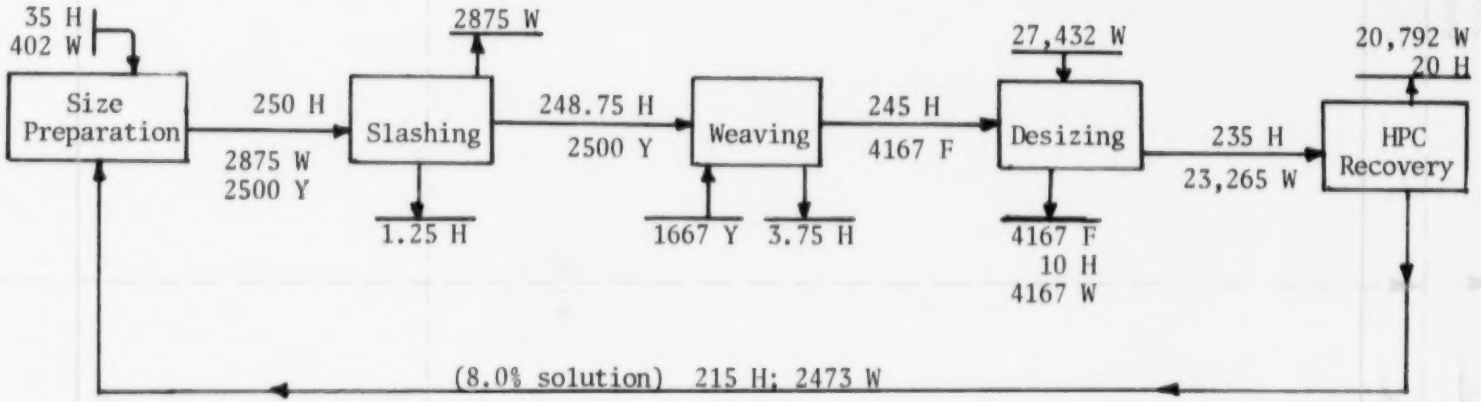

FIGURE 8. MATERIAL BALANCES FOR THERMAL PRECIPITATION RECLAMATION SYSTEM

$0.70/pound for PVA, $2.00 for HPC and $0.07 for starch), the initial investment for size is $17,500 for PVA for ultrafiltration, $50,000 for HPC for thermal precipitation and $6,250 for a PVA/starch mixture not being recovered.

The return on investment is good for a size reclamation system. The costs shown in Table 23 give a ROI of 17.1% for ultrafiltration and 42.0% for thermal precipitation. The payout time in years is much smaller for a thermal precipitation system (2.04 years) than for an ultrafiltration system (3.74 years) because of the lower capital investment for thermal precipitation.

Details of the economics for an ultrafiltration size reclamation system are shown in Table 24. Equipment costs were obtained from literature published by Abcor, Inc., and installation cost was calculated at 70% of equipment cost which was the actual percentage for an ultrafiltration system installed by a major textile company.

Details of the economics for a thermal precipitation size reclamation system are shown in Table 25. The equipment costs are based on the system shown in Figure 8 which requires the equipment listed in Table 26.

It is significant that the fixed capital investment required for size reclamation by thermal precipitation is about one-third of that required by ultrafiltration.

Costs for labor, maintenance materials and taxes and insurance were calculated at the same percentages for thermal precipitation and ultrafiltration. Pumping power requirements are considerably lower for thermal precipitation than for ultrafiltration because the latter offers considerable resistance due to the membranes. This is important since energy costs are expected to continue to increase indefinitely.

The cost of energy for hot water is much higher for ultrafiltration than for thermal precipitation. No credit has been allowed for recycling of desize washwater in an ultrafiltration system. This may be possible and, if done, several thousand dollars in energy savings would be realized. However, the ultrafiltration systems operating in the U.S. today do not return the water to the desize washer after PVA removal.

Wastewater treatment costs will be reduced dramatically if a size reclamation system is used regardless of whether the system is based on thermal precipitation or ultrafiltration. The magnitude of reduction of BOD and COD of a plant will depend on what type of size the mill had been previously using. If the mill processes fabrics containing starch with a BOD of about 50%, the BOD reduction achieved by size reclamation would be dramatic. If the mill processes fabrics containing primarily synthetic sizes such as polyvinyl alcohol or carboxymethylcellulose which have low BOD's but relatively high COD's, the pollution control benefits will be mainly in lower solids content and COD of the wastewater.

A major item in the cost of the slashing process is the cost of the size

TABLE 24. ECONOMICS OF SIZE RECLAMATION BY ULTRAFILTRATION[1]

CATEGORY	COST (1977)
INVESTMENT	
Ultrafiltration System Cost[2]	$463,400
Field Installation (70% of System Cost)	324,400
Capital Investment	787,800
Initial Size (25,000 lbs. @ $0.70/lb.)	17,500
Total Capital Investment	$805,300
ANNUAL OPERATING COSTS	
Pumping Power[3]	$ 7,530
Operating and Maintenance Labor[4]	45,000
Supervisory Labor[5]	7,000
Maintenance Materials (2% of UF System Cost)	9,270
Taxes and Ins. (2% of Total Installed Cost)	15,760
Membrane Replacement[6]	86,170
Size (PVA) Replacement[7]	251,900
Wastewater Treatment[8]	20,000
Energy for Hot Water[9]	75,000
Depreciation (10 years straight line)	78,780
Total Annual Operating Costs	$596,410

[1]Based on Abcor, Inc. data (Reference #4).
[2]$305,000 (in 1976 dollars) for 43,200 gallons per day system adjusted to 72,000 gallons per day in 1977 dollars.
[3]$4,520 for 43,200 gallons per day adjusted to 72,000 gallons per day.
[4]One (1) operator per shift @ $5/hr. + 20% overhead.
[5]One engineer @ 1/3 time
[6]$51,700 for 43,200 gallons per day adjusted to 72,000 gallons per day.
[7]20% loss of PVA replaced @ $0.70/lb.
[8]For 1983 BATEA. (Reference #3).
[9]To heat 3,000 gpm from 70°F to 205°F.

TABLE 25. ECONOMICS OF SIZE RECLAMATION USING THERMAL PRECIPITATION.

CATEGORY	COST (1977)
INVESTMENT	
Equipment[1]	$ 80,000
Instrumentation, controls, piping, electrical and installation[2]	78,400
TOTAL DIRECT COSTS (TDC)	$ 158,400
Engineering and Construction (20% of TDC)	31,580
TOTAL DIRECT AND INDIRECT COSTS	$ 190,080
Contractor's Fee and Contingency (12% of TDC and Indirect Cost)	22,800
Capital Investment	212,590
Initial Size (25,000 lbs. @ $2/lb.)	50,000
TOTAL CAPITAL INVESTMENT	$ 262,590
ANNUAL OPERATING COSTS	
Pumping Power (20 HP)	$ 2,150
Labor (Operating, maintenance, supervisory)	52,000
Maintenance Materials (2% of TDC)	3,200
Taxes and Insurance[3]	4,200
Energy for hot water and cooling water	21,000
Size (HPC) replacement[4]	507,500
Wastewater Treatment	14,000
Depreciation (10 years straight line)	21,300
TOTAL ANNUAL OPERATING COSTS	$ 625,350

[1]As specified in Table 26.

[2]98% of equipment cost.

[3]2% of fixed capital investment excluding initial size material purchased.

[4]14% loss of HPC replaced @ $1.75/lb.

TABLE 26. SPECIFICATIONS FOR EQUIPMENT FOR THERMAL PRECIPITATION RECOVERY SYSTEM

ITEM	DESCRIPTION	COST (1977)
T1	Desize washwater holding tank 75,000 gallon capacity, carbon steel	$ 25,470
T-2A,B	Separation tanks, jacketed, 4,500 gallon capacity, carbon steel	28,050
T-3	Concentrate storage tank, 1000 gal. capacity, carbon steel	2,405
T-4	Reclaimed water storage tank 10,000 gal. capacity, carbon steel	8,625
P-1A,B	Pump, 300 gpm, 50' head with motor (with spare)	2,725
P-2A,B	Pump, 300 gpm, 50' head with motor (with spare)	2,720
P-3A,B	Pump, 10 gpm, 50' head with motor	1,720
P-4A,B	Pump, 75 gpm, 50' head with motor (with spare)	1,550
P-5A,B	Pump, 75 gpm, 50' head with motor (with spare)	1,550
P-6A,B	150 gpm, 50' head with motor	2,090
E-1	Heat Exchanger, 50 ft.^{2}	1,475
E-2	Heat Exchanger, 70 ft.^{2}	1,620
	TOTAL EQUIPMENT COST	$80,000

material itself. Conventional slashing systems for polyester/cotton blend yarns often use a mixture of polyvinyl alcohol and starch. If a 30/70 weight percent PVA/starch mixture is applied at a level of 6% of fabric weight, then the size which is removed in the desizing process and subsequently discharged in the plant waste represents a loss of $460,000 per year. If an ultrafiltration reclamation system is used, only pure PVA is used because starch size is not recoverable. PVA has been applied to textile yarns and subsequently recovered by ultrafiltration at a recovery level of 80%. Thus, the PVA loss amounts to $251,000. If HPC is used and subsequently recovered by thermal precipitation at a recovery level of 85%*, the replacement HPC costs $507,500. The HPC replacement cost is computed at a cost of $1.75 per pound of HPC. The current cost of HPC is about $2.00 per pound, but according to the chemical supplier HPC would sell for the lower price of $1.75 per pound if it were bought in large volumes by the textile industry.

In summary, size reclamation systems promise a high return on investment in addition to the pollution control benefits which can be realized. In the case of reclamation of HPC by thermal precipitation, annual savings of $108,000 would be achieved on an investment of $262,000 giving a ROI of 42.0%. In the case of reclamation of PVA by ultrafiltration, annual savings of $137,000 would be achieved on an investment of $805,000 giving a ROI of 17.1%.

COST OF ENERGY

There are several steps in sizing, desizing and size reclamation which require thermal and electrical energy. The water heating and drying requirements are the most energy-consuming. The requirements for heat are the same in slashing regardless of whether a conventional size or a recoverable size is used. However, in conventional desizing or desizing of polyvinyl alcohol for reclamation by ultrafiltration, the water must be heated to about 200°F from the incoming temperature of 80°F in order to achieve efficient size removal from the fabric. This requires about 2.73×10^6 BTU's per hour where 4167 pounds of fabric is processed. On the other hand, desizing and size reclamation using a thermal precipitation system requires only that the water be heated to 130°F. This requires only 1.16×10^6 BTU's per hour. Therefore 1.57×10^6 less BTU's per hour are used in the thermal precipitation system than in the ultrafiltration system giving savings of about $50,000 per year at a steam cost of $4.55 per 1000 pounds. In addition, the electrical energy for pumping the desize washwater costs about $5,000 per year more for ultrafiltration than for thermal precipitation because of the resistance to flow contributed by the ultrafiltration membranes.

*The recovery level of 86% is based on HPC losses of 0.5% at slashing, 1.5% at weaving, 4.0% residual on the fabric and 8.0% in the reclamation system. Laboratory studies have demonstrated that this recovery level is obtainable.

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ENGLISH TO METRIC CONVERSION FACTORS

1 inch (in) = 2.540 centimeters (cm)

1 foot (ft) = 30.48 cm

1 yard (yd) = 0.914 meters (m) = 91.44 cm

1 cm = 0.394 in

1 pound (lb) = 0.454 Kilograms (Kg) = 453.6 grams (g)

1 Kg = 2.205 lb

1 gallon (gal) = 3.785 liters (l)

1 l = 0.264 gal

°F = 9/5 (°C) + 32

°C = 5/9 (°F - 32)

GLOSSARY

Certain terms used in this report have special meanings in the field of textiles. These definitions are provided here to assist readers who do not have a textile background.

Break factor - the numerical product of the breaking strength in ounces of a yarn and the yarn's cotton count.

Bust, lease or split rod - stainless steel rods used in a slasher to split or separate the yarns which have been stuck together with sizing material.

Cotton count - A number expressing a yarn's length per unit weight where the number represents the "count" of 840-yard lengths that weigh exactly one pound.

End - a single yarn, usually in the warp.

Filling - the widthwise yarns in a woven fabric; also weft yarns.

Harnesses - frames that contain the needles for control of warp yarns in a loom.

Pick - a single filling or weft yarn in a woven fabric.

Reed - that part of a loom that dictates the spacing of warp ends to determine ends per inch in the fabric.

Splitting - separation of sized warp yarns by action of the bust or lease rods.

Warp - the lengthwise yarns in a woven fabric.